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Development and characterization of a peanut-shell based activated carbon and the outcomes of a hands-on approach to chemical education

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Development and characterization of a peanut-shell based activated carbon and the outcomes of a hands-on approach to chemical education

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Heavy metals are a recognized toxic environmental contaminant, even at very low concentrations. There have been well-known events in the last decade within the US of high amounts of lead in the drinking water supplies of cities, leading to detrimental effects within its population. Ways have been found to remove this metal, and others, from water with expensive adsorbents. The aim of the first part of this research was to create an inexpensive adsorbent from a waste material and modify it in such a way that it would be adept at removing heavy metals from water.

In Chapter I, we were able to remove lead, copper, and cadmium using our peanut shell-based activated carbon, getting a high amount of metal adsorption when the activated carbon was activated with phosphoric acid, pyrolyzed, and then cooled under a nitrogen atmosphere. The activated carbon was characterized and found to have a BET surface area of 781 m^2g^{-1} and a Langmuir maximum isotherm capacity of 100.2 mg/g. By using the data

obtained in this work, it could lead to the development of further economically made adsorbents to be used to provide more people with clean drinking water.

The second part of our work focused on the benefit of a hands-on approach to chemical education. In Chapter IV, we discuss the development and implementation of our NSF-funded summer research experience for undergraduates program, as well as the student-reported results from their 10-week research experience. These surveys showed consistent self-reported growth among the student cohort in the skill sets that were focused on during the program.

Chapter V focuses on the development, application, and analysis of results for a novel home-based laboratory component for a semester-long organic chemistry course. It featured 12 lab activities: 8 hands-on experiments and 4 online modeling exercises. By developing and sharing this off-campus approach, we hope to provide an option for other universities that are looking for at-home laboratory experiences for their own students.

Overall, we found that these approaches to experiencing chemistry in a hands-on way were beneficial to students and provided them with a greater interest in chemistry.

DEDICATION

This work is dedicated to my late grandmother, Doris Pitre

who always believed in me and loved me unconditionally

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CHAPTER I

THE EFFECTS OF HEAVY METAL CONTAMINANTS IN WATER AND THE USE OF ACTIVATED CARBON FOR REMEDIATION

1.1 Contamination of Water

Water is one of the most precious resources on Earth, because although 70% of the planet is covered in it, according to the Environmental Protection Agency (EPA) less than 1% of it is accessible for human use[1]. Because of the scarcity of fresh and available water, it is vital that as much as possible is done to keep water free from contamination. There are many types of contaminations that can impact water sources, and one that has been brought to the public spotlight more often in recent years is the contamination of water by heavy metals.

Historically, there have been many documented cases of heavy metal contamination in water systems, perhaps the most recent well-publicized case being the 2014 matter of Flint, MI, in which the city's water supply switched providers and lead was found to be leaching from the corroded pipes into the water[2-4]. Other lesser-known cases have been reported as well, such as in

Washington, DC, where a change in disinfectant in 2000 led to lead leaching[5,6], extensive arsenic contamination of groundwater in areas of Asia[7], as well as a wide variety of heavy metals released into water sources due to abandoned coal mines in China[8].

1.2 Effects and Consequences of Contaminated Water

When heavy metal contamination catastrophes happen and the levels of heavy metals in water systems are found to be too high, further work must be done to find the far-reaching consequences of those contaminations, such as lasting effects on the environment or on the population. Examples of these effects are numerous such as the San Francisco Bay surface waters having a much higher concentration of lead than the surrounding water due to runoff from California's landmass[9] and in Nantong, China, elevated levels of lead were found throughout the environment, even in the dust and soil[10].

Further reaching effects can be those which have been tied directly to impacts on the population of the areas where contamination has occurred. On Sand Island, Midway Atoll, albatross chicks were found to have been exposed to lead through picking at and ingesting paint chips, leading to lead poisoning in the birds' population[11]. In the previously mentioned Washington, DC lead crisis, fetal death rates peaked immediately following in 2001[12], and the lead blood level in children increased more than four times[5].

1.2.1 Reduction of Effects after Water Remediation

Effects on the population rise as contaminant concentrations rise, the inverse is often true as well. Reducing the amount of contaminants making their way into the water systems of populations can lead to a reduction in contaminant concentrations within the population[13], as seen in Nantong, China, when lead control was enacted and lead concentrations in environmental and in biological samples began to fall[10]. Similarly, in New Orleans, Louisiana, soil lead concentrations fell due to the tragedy of Hurricanes Katrina and Rita, which was followed by a reduction in blood lead concentrations in children[14].

1.2.2 Efficient Methods of Heavy Metal Removal

Because heavy metals are such a detriment to the population when they are present, it is imperative that efficient ways are found for their monitoring[15], detection[16], and removal. The EPA has frequently used a coagulation/filtration method for the removal of various heavy metals, such as injecting $FeCl_3$ into a system to form particles that could then be removed by pressure filtration. They have also employed various adsorptive medias, such as iron-based medias in the case of arsenic removal from water systems[17].

As it is such an important field of study, much work has been done in the area of water remediation where heavy metals are concerned, such as arsenic removal being performed both by cactus mucilage[18] and bauxsol[19]. Bauxsol has potential to remove multiple metals and was used to remove lead, copper, and

zinc from water[19]. Diatomite also demonstrated the ability to remove multiple metals[20]. Nanoparticles are another viable area of this field, as both the use of metal-organic framework beads containing inorganic nanoparticles[21] and magnetic nanoparticles coated with humic acid[22] have shown that they are well-able to remove a variety of heavy metals from water samples.

1.3 Activated Carbon

Adsorption is a highly used and efficient method for removing heavy metals from water, and one type of adsorbent that has been used in recent years for the removal of heavy metals, along with other contaminants, from water is activated carbon, or biochar, as it is also known. Biochar is a carbonaceous byproduct made by the pyrolysis of biomass, with a huge variety of biomasses already in use[23].

1.3.1 Common Biomasses

The biomasses commonly used to make biochar range widely and can be utilized based on their ability to produce biochars that adsorb certain compounds or even by their available surface area. Waste products, such as orange peels[24], coffee grounds[25], pig manure[26], wheat straw[26], invasive plants[27], hickory wood[28], and as in our own work, peanut shells, can be used as biomass. These waste products have the advantage of being cost-effective, readily available, and generally of little use otherwise, which make for sustainable adsorbents[29–31].

Table 1.1 gives a shortened list of common biomasses used to make activated carbon and biochar, as well as the conditions used to make them.

Table 1.1 Common biomasses and their preparation conditions[32]

Biomass	**Pyrolysis Temperature (°C) and Time**		**Biomass**	**Pyrolysis Temperature (°C) and Time**
Corncob	500/700, 2 h		Bamboo	700, 3 h
Pine Cone	500, 2.5 h		Pig Manure	400, 1 h
Switch Grass	425, 1 min		Straw	400-525, 10 h
Wheat Husk	500, 20 min		Spruce	400-525, 10 h
Rice Straw	250-450, 2-8 h		Wood Chips	620, 20 min
Douglas Fir Wood	623-823, 0.5 h		Sewage Sludge	600, 20 min
Sugarcane Straw	700, 1h		Sugar Beet Tailings	450, 6 h

1.3.2 Advantages for Adsorption

There are many features of activated carbon and biochar that make them advantageous adsorbents, besides their low-cost and high availability. These adsorbents have unique surface properties, including a high surface area to weight ratio and a spectrum of possible surface functional groups[28,33], all of which can vary depending on the type of biomass and the parameters chosen during its preparation[34,35].

A high surface area to weight ratio is dependent in the material used, as more porous materials have more area for adsorption to happen, but it can also be affected by temperature of pyrolysis[36,37] and activation method[38]. For example, a biochar made from cotton straw had a specific surface area of 158.8 m^2 g^{-1} when a temperature of 850 C was used, versus 3.9 m^2 g^{-1} at 450 C[39].

A biochar having high porosity and high surface area leads to greater efficiency for adsorption[40–42], as less char is needed to adsorb the analyte in question. High porosity size is also needed if adsorption of larger molecules is the target, or else only the outside surface of the adsorbent would be available for adsorption[43].

1.3.3 Common Functionalizations and Applications

Not only do the pyrolysis parameters influence the properties of the adsorbent, but modifications can also be made to the materials before and after pyrolysis to further affect the adsorption properties. There are many examples from literature where these modifications led to improvements in the adsorbent.

Utilization of ozonization led to a tenfold increase of cation exchange capacity in one study[44], with a high CEC being an indicator of the biochar's effectiveness in soil amendment. Many studies[29,31] have magnetized the adsorbents used in their work, impregnating the char pre-pyrolysis with iron to form iron oxides on its surface, leading to faster and easier adsorbent removal[31] as well as opportunities for unique teaching laboratory experiments[29].

Zhang et al.[45] pyrolyzed their biomass matrix after soaking them in a $MgCl_2$ mixture, forming MgO particles within the adsorbent. MgO particles have a positive surface charge, indicating an increased ability to adsorb anions, and their adsorbents showed a capacity to efficiently adsorb phosphates and nitrates.

An $AlCl_3$ solution was used pre-pyrolization to form an AlOOH/biochar nanocomposite, leading to the adsorbent being capable of adsorbing arsenic, methylene blue, and phosphates by increasing the adsorbent's capabilities through the use of the AlOOH nanoparticles[46]. Steam activation has been used by multitudes of groups, due to its ability to increase the adsorbent's surface area, pore space and size, and absorptive capacity for smaller molecules[27,47].

1.3.4 Common Types of Analyte Adsorption

Because activated carbons and biochars can vary widely depending on the biomass used, pyrolysis parameters such as temperature and time, as well as functional modifications used for their preparation, it is not surprising to find that the range of analytes capable of being adsorbed by these materials is just as diverse. Analytes that have been successfully removed using these adsorbents include phosphates[45,48], nitrates[45], organic contaminants[27,29,49], and CO_2[28,50]. The effective removal of these analytes, along with many more, lays the foundation for further research in the field. Table 1.2 provides a list of biomasses used for the successful removal of various contaminants.

Table 1.2 Contaminants removed by varying types of biochar[23]

Contaminant	Original Biomass		Contaminant	Original Biomass
Atrazine (herbicide)	Dairy Manure		Arsenic	Hard Wood
Tetracycline (antibiotic)	Rice Husk		Mercury	Soybean Stalk
Naphthalene	Orange Peel		Pentachlorophenol (pesticide)	Bamboo
Nitrobenzene	Pine Needles		Polycyclic Aromatic Hydrocarbons	Sewage Sludge
Chromium	Oak Wood		Trichloroethylene	Peanut Shell

Much work has also been done in using these adsorbents for the removal of heavy metal contaminants in various environments. As previously mentioned, heavy metals left in the environment can and do have detrimental effects on the affected population. Because activated carbon and biochar are so readily available and cost-effective, they are a prime candidate for helping to remove these metals from the environment.

Biochar made from rice straw that was grown in cadmium-polluted water has been used to further remove cadmium from irrigation ponds[51], ZnS nanocrystals were added to magnetic biochar and drastically increased its capacity to adsorb lead from contaminated water[52], biochar coated with MoS_2 was shown to exhibit highly selective lead adsorption even when in the presence of other metals such as cobalt and cadmium[53]. A variety of magnesium-loaded

adsorbents have been successfully used for the adsorption of cadmium, lead, and copper[54]. These are just a few of the many impressive examples available that exhibit the value that these adsorbents have on environmental contaminant clean-ups.

Efficiency in other water remediation besides that involving heavy metals has also been demonstrated by both activated carbons and biochars. Biochar has been used in wastewater treatments as an additive to sludge to affect anaerobic digestion[55], several biochars were modified to have increased hydrophobicity and further displayed efficiency at removing oil in simulated oil spills[56], both activated carbon and biochar were used to remove organic micropollutants in wastewater treatment facilities[57], and biochar has even been used as a basis for solar adsorbers in steam generation and clean water production[58].

1.4 Research Objectives

The objective of our study was to create a high-performing adsorbent by starting with an inexpensive waste material (peanut shells), modifying the biomass, and pyrolyzing the material in order to form an activated carbon. Once formed, the activated carbon was characterized to determine its optimal adsorption parameters, and those parameters were then used in the adsorption of multiple heavy metals. The results of this adsorbent will be discussed in chapter 2.

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CHAPTER II

HEAVY METAL REMOVAL USING NITROGEN-COOLED PEANUT SHELL BASED ACTIVATED CARBON

2.1 Abstract

Activated carbon was produced from peanut shells soaked in H_3PO_4 and then heated for 1 h at 850 °C followed by cooling under nitrogen. The activated carbon (PSAC) was used to remove lead, cadmium, and copper from synthetic wastewater containing them. Experiments were performed to find the optimal adsorption time, pH, and PSAC dosage, and these optimized parameters were used for further adsorption studies. This PSAC had a point of zero charge of 2.05, Langmuir maximum isotherm capacity of 100.2 mg/g and BET surface area of 781 m^2g^{-1}. Surface morphology was characterized by SEM and TGA.

2.2 Introduction

Recent and ongoing events in Flint, MI[1-3] have brought to light that heavy metal contamination in water systems is a serious environmental concern that can lead to detrimental health effects in humans[4], especially children[5]. Metals such as lead, copper, and cadmium that have found their way into the water

systems via channels such as pipe leaching[6,7] and industry runoff[8-11]. The EPA[12] has reported the satisfactory level of lead in water as zero, with treatment measures[13] in place to take action should the level reach 0.015 ppm. The EPA's acceptable levels of copper and cadmium in drinking water are 1.3 ppm and 0.005 ppm, respectively[12].

Many adsorbents have been developed to remove heavy metals from water, including limestone[14], tea waste[15], and many other agricultural wastes[16]. However, activated carbon has shown to be highly effective and is used industrially for gas[17] and water purification[18]. Because of its high porosity and large surface area, activated carbon is suitable for the adsorption of unwanted pollutants, such as heavy metals, from contaminated samples. Activated carbon can be made from many different agricultural wastes[19-23], including peanut shells, which have been reported as having favorable adsorptive capabilities for heavy metals[24-29]. Our decision to use peanut shells as a biomass was to allow a waste product to be converted into a product that can be used in water remediation, utilizing an otherwise unused material that would normally be disposed of.

Activated carbons have been produced under various atmospheres such as air, nitrogen, and steam, as well as activated by various acid and base treatments[30-32]. Here we pyrolyzed peanut shells chemically activated with H_3PO_4 before heating under limited oxygen and afterwards cooling under nitrogen. H_3PO_4 was used as an activating agent because in previous literature[33,34], it has been found to increase the adsorption of metals onto the pyrolyzed material. The

resulting PSAC was characterized and its adsorption properties for lead, cadmium, and copper were determined. The optimum parameters such as adsorbent dose, pH, and adsorption time were determined.

2.3 Experimental

2.3.1 Reagents and Equipment

All chemicals and reagents used were of AR or GR quality, and purchased from Sigma-Aldrich (St. Louis, MO). Stock solutions were made by dissolving lead nitrate, copper sulfate, or cadmium nitrate in deionized water from a Millipore-Q water system. Water pH adjustments were made by using either 0.1M HNO_3 or 0.1M NaOH, and pH measurements made by a pH meter (Hanna instrument HI 2211 pH/ORP meter). Measurements of the concentrations of the lead, cadmium, and copper solutions were determined using a Flame Atomic Absorption Spectrophotometer (AAS) (Shimadzu AA-7000). All studies requiring equilibration of adsorbents were performed in an orbital shaker (ThermoForma Orbital Shaker).

2.4 Activated Carbon Preparation

Raw peanuts were purchased, shelled, and the shells collected for further use. The shells were washed with deionized (DI) water to remove debris and broken into smaller pieces approximately 20 mm in diameter before drying in an oven at 110 °C until a constant weight was reached. The shells were then further

crushed into smaller pieces approximately 10 mm in diameter, mixed with a 1:1 mass ratio of 85% H_3PO_4, and allowed to soak for 1 h for activation. After soaking, the shells were placed within a lidded crucible to limit oxygen exposure, and then placed in a pre-heated muffle furnace for at 850 °C. After 1 h at 850 °C, the hot activated carbon was immediately transferred to a gas tight chamber where a constant flow (20 mL/min) of nitrogen passed through the product for approximately 1 h until the activated carbon cooled to room temperature.

After cooling, the activated carbon was washed via filtration with DI water until a pH of 6 was reached in the effluent water, and then the carbon was dried in an oven at 110 °C for 24 h. After drying, the activated carbon was ground and sieved to a particle size range of 100 to 600 μm and stored for further use, and this peanut shell activated carbon is designated as PSAC.

2.5 Adsorbent Characterization

The point of zero charge (PZC) was determined by adjusting pH using 0.1M HNO_3 and 0.1M NaOH solutions to values ranging from 2 to 11. DI water solutions (10 mL) were pH adjusted before addition of 0.002 g/mL of PSAC, followed by shaking for 24 h. The PSAC was removed by filtration using filter paper and the pH of the filtrate was measured. The pH of the initial solution was plotted against the pH of the final solutions, and the intersection of the lines gave the PZC for the char.

PSAC adsorbent morphologies were imaged using scanning electron microscopy (JEOL JSM-6500F FE-SEM, operated at 5 kV). A carbon stud was attached to carbon tape, then sputter-coated under argon with a 25 nm layer of platinum using an EMS 150T ES sputter coater. Surface area and pore size of the adsorbent (PSAC) was analyzed by N_2 adsorption isotherm at 77 K using a Brunauer-Emmett-Teller (BET) analyzer (MicroActive for TriStar II Plus 2.03). Thermogravimetric analysis (TGA) of PSAC was performed using a TA Q-50 thermogravimetric analyzer.

2.6 Sorption Studies

2.6.1 Adsorbent Dosage Optimization

Varied doses of PSAC were added to solutions containing measured concentrations of lead nitrate, cadmium nitrate, or copper sulfate. Each replication was performed using 10 mL of 100 ppm $Pb(NO_3)_2$, 100 ppm $Cd(NO_3)_2$, or 100 ppm $Cu(SO_4)_2$ to determine the optimum amount of activated carbon for adsorption. For lead optimization, a PSAC dose range of 0.015 g to 0.100 g was used. For cadmium optimization, a dose range of 0.015 g to 0.035 g was used. Copper optimizations employed a dosage range of 0.010 g to 0.030 g. All dosage experiments were shaken for 17 h at 25 °C and 175 rpm, and each experiment was performed in triplicate with error bars reporting standard deviation of the triplicates. After shaking, the contents of the tubes were filtered through

Whatman No.1 filter paper and the filtrate saved for later metal concentration analysis.

The data was analyzed to find the best ratio of adsorbent used vs. specific adsorption. The optimal PSAC doses for lead, cadmium, and copper adsorption were found to be 0.002 g/mL, 0.0015 g/mL, and 0.001 g/mL, respectively. Figure 2.1 shows the specific adsorption varying by dosage amount for each metal.

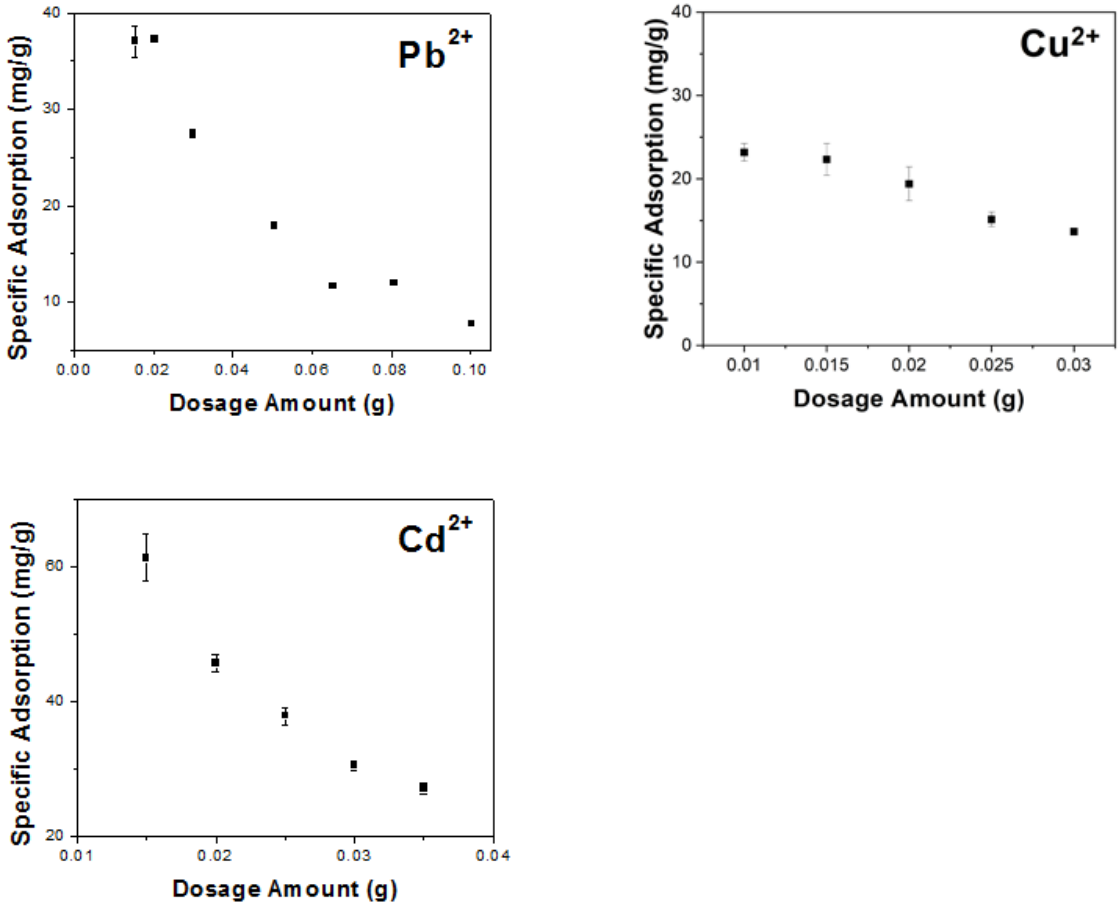

Figure 2.1 Dosage optimization of lead, copper, and cadmium

2.6.2 pH Optimization

PSAC lead adsorption was studied over a pH range of 2-6. PSAC (0.02 g, or 0.002g/mL) was added to 10 mL of the pH-adjusted 100 ppm $Pb(NO_3)_2$ solutions. These solutions were shaken at 25 ºC and 175 rpm for 17 h, then filtered through Whatman No.1 filter paper. The filtrate was stored in sealed test tubes for later lead analysis.

2.6.3 Kinetic Studies

Kinetic studies were performed for each metal. PSAC (0.002 g/mL, 0.0015 g/mL, or 0.001 g/mL) was added to 10 mL of a previously prepared metal solution containing 100 ppm of either lead, cadmium, or copper, respectively. The PSAC/metal solutions were shaken at 25 ºC and 175 rpm, for specific time intervals from 5 min to 24 h followed by filtration through Whatman No.1 filter paper. The filtrate was stored in sealed test tubes for later analysis.

2.6.4 Isotherm Studies

In order to fit adsorption isotherm data to known models and equations, isotherm experiments were performed at multiple temperatures. PSAC (0.002 g/mL) was added to a series of 10 mL of previously prepared lead nitrate solutions with concentrations ranging from 40 to 450 ppm. The solutions were shaken for 24 h at 175 rpm at 25, 35, and 45 ºC. Then the solutions were filtered through Whatman No.1 filter paper and the filtrate saved for metals analysis.

2.7 Results and Discussion

2.7.1 Characterization of PSAC

The point of zero charge (PZC) of PSAC is at pH 2.05. Below this pH PSAC will be positively charged and above this pH, the surface will be negatively charged, likely favoring attraction of Pb^{2+}, Cu^{2+}, and Cd^{2+} cations.

Table 2.1 Determination of Point of Zero Charge (PZC)

Points of reference				**PSAC**		
$pH_{(initial)}$		$pH_{(final)}$		$pH_{(initial)}$		$pH_{(final)}$
0		0				
2.04		2.04		2.04		2.05
3.01		3.01		3.01		2.91
3.95		3.95		3.95		3.11
4.93		4.93		4.93		3.12
6.02		6.02		6.02		3.21
6.94		6.94		6.94		3.34
8.01		8.01		8.01		3.00
9.05		9.05		9.05		3.15
10.00		10.00		10.00		3.74
11.01		11.01		11.01		4.38

The point of zero charge for the peanut shell activated carbon is 2.05.

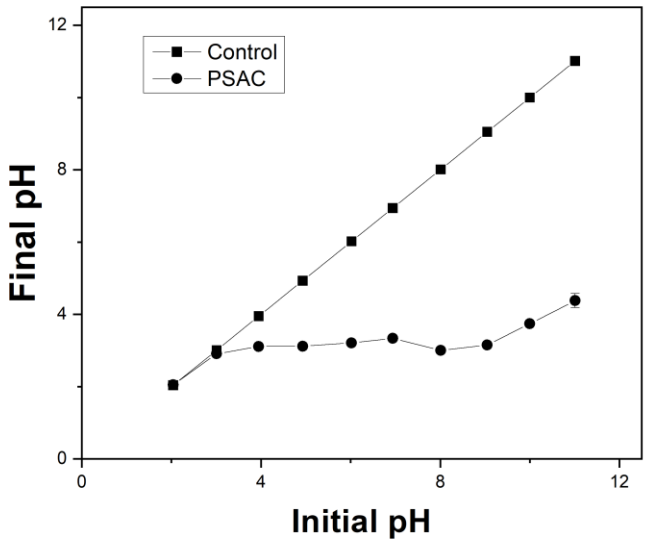

Figure 2.2 Point of zero charge data plot

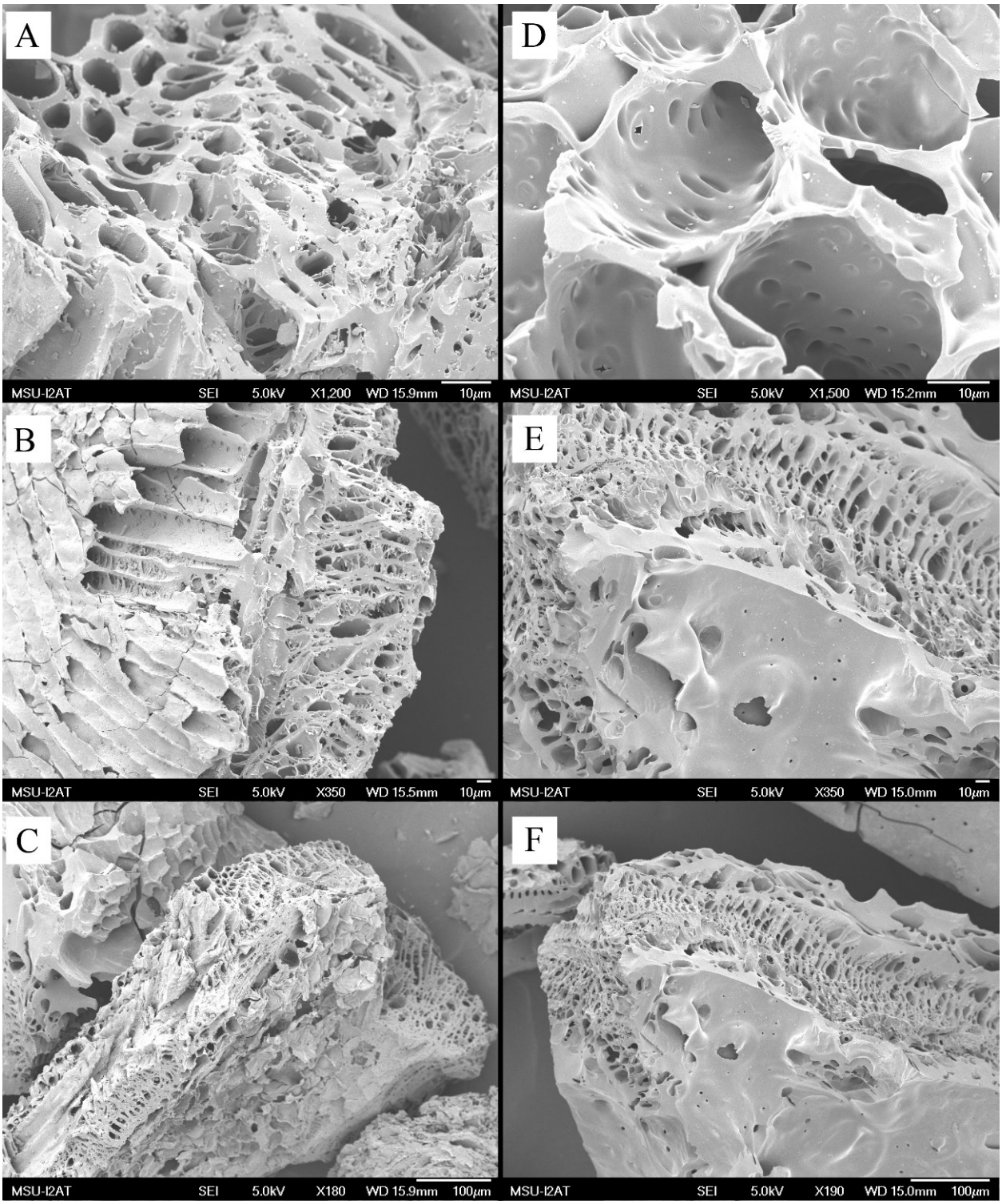

Figure 2.3 SEM imaging of PSAC

In Figure 2.3, SEM images of PSAC are shown at varying magnifications). Images A-C display PSAC before adsorption experiments were performed. The macropores in the activated carbon can be seen and noted for their honeycomb-like surface. D-F are SEM images of PSAC after adsorption of lead.

Figure 2.4 includes the TGA decomposition curve of PSAC, demonstrating the change in sample mass that occurred when the sample was heated from ambient 20 °C to 1000 °C.

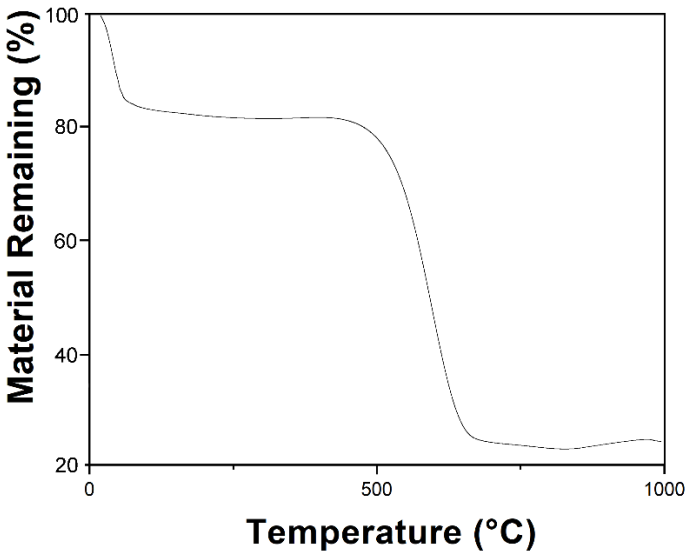

Figure 2.4 TGA decomposition curve of PSAC

Table 2.2 Surface area characteristics of PSAC adsorbent

Parameters			
S_{BET}			781
(m^2 g^{-1})			
Pore volume			0.432
(cm^3 g^{-1})			
Pore size (Å)			22.2

Table 2.2 provides information from the BET analysis of PSAC, as discussed in section 2.5.

2.7.2 pH Optimization and its Effect on Adsorption

Adsorption variations with pH were studied using the previously prepared 100 ppm lead nitrate solutions adjusted to pH values ranging from 2 to 6. PSAC (0.002 g/mL) was shaken with 10 mL of lead nitrate solution. Lead adsorption peaked at pH 5. Above a pH of 6, the lead began precipitating instead of adsorbing to the activated carbon. The amount of lead adsorbed ranged from 28.9 mg/g at a pH of 2 to 37.3 mg/g at a pH of 5 (Figure 2.5). A pH of 5 was used for all further experiments.

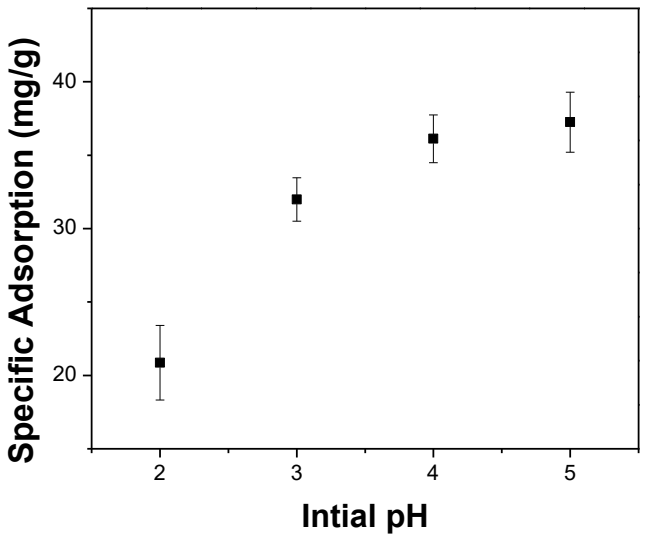

Figure 2.5 Effect of pH on the adsorption of lead onto PSAC.

2.7.3 Adsorption Isotherms

The adsorption of lead onto PSAC was analyzed at three temperatures, 25 °C, 35 °C, and 45 °C and that data fit to Langmuir[35] and Freundlich[36] isotherm models to determine the maximum adsorption capacity of PSAC. Figure 2.6 shows the experimental adsorption data fitted to the Langmuir and Freundlich isotherms using Origin 2017. The R^2 values for the Langmuir model support the assumption of monolayer lead adsorption onto the PSAC. The Langmuir maximum adsorption capacities for PSAC were 100.2, 87.8, and 84.9 mg/g at 25, 35 and 45 °C, respectively. These results are displayed in Figure 2.6.

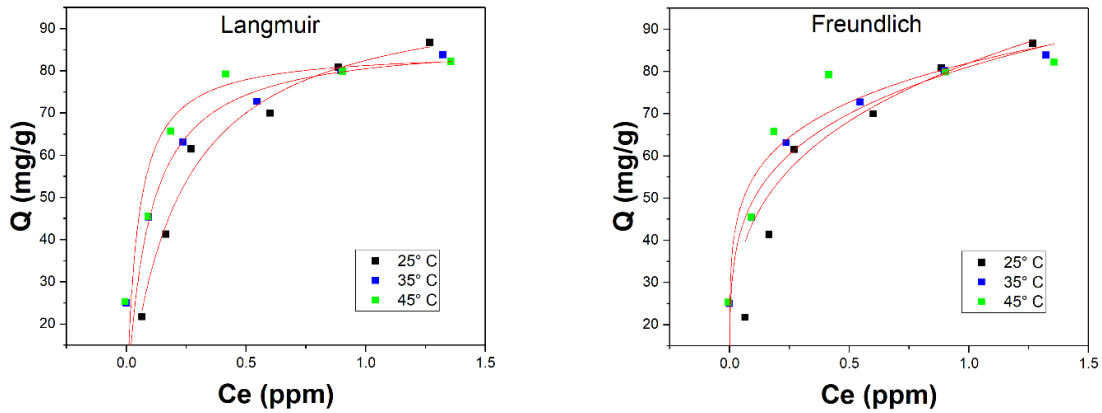

Figure 2.6 Adsorption of lead onto PSAC at 25, 35 and 45 °C, fitted using Langmuir and Freundlich isotherm models. Ce is concentration of lead at equilibrium (mg/L). Q is the lead concentration on adsorbent (mg L/g PSAC).

Table 2.3 Isotherm parameters for PSAC

Isotherm	**Parameters**	**25 ˚C**	**35 ˚C**	**45 ˚C**
Langmuir	Q^0 (mg/g)	100.2	87.8	84.9
	b	4.64	11.0	22.3
	R^2	0.977	0.690	0.999
Freundlich	K_f (mg/g)	82.00	81.1	82.00
	n	3.78	4.70	5.75
	R^2	1.00	0.680	0.610

Table 2.3 includes the parameters used for fitting the isotherm models. The values for Langmuir and Freundlich can be compared for each temperature.

2.7.4 Adsorption Kinetics

Figure 2.7 shows the amount of lead, copper, or cadmium adsorbed onto the PSAC surface at each sampled time interval, ranging from 5 to 1440 min (24 h). The adsorption of each metal occurred quickly in the first 3 to 5 h and then more slowly afterwards. Cd^{2+} could still be slowly increasing after 24 h, but equilibrium appeared to have been reached by 17 h for Pb^{2+} and Cu^{2+}. A period of 24 h was used for each metal to ensure optimum adsorption.

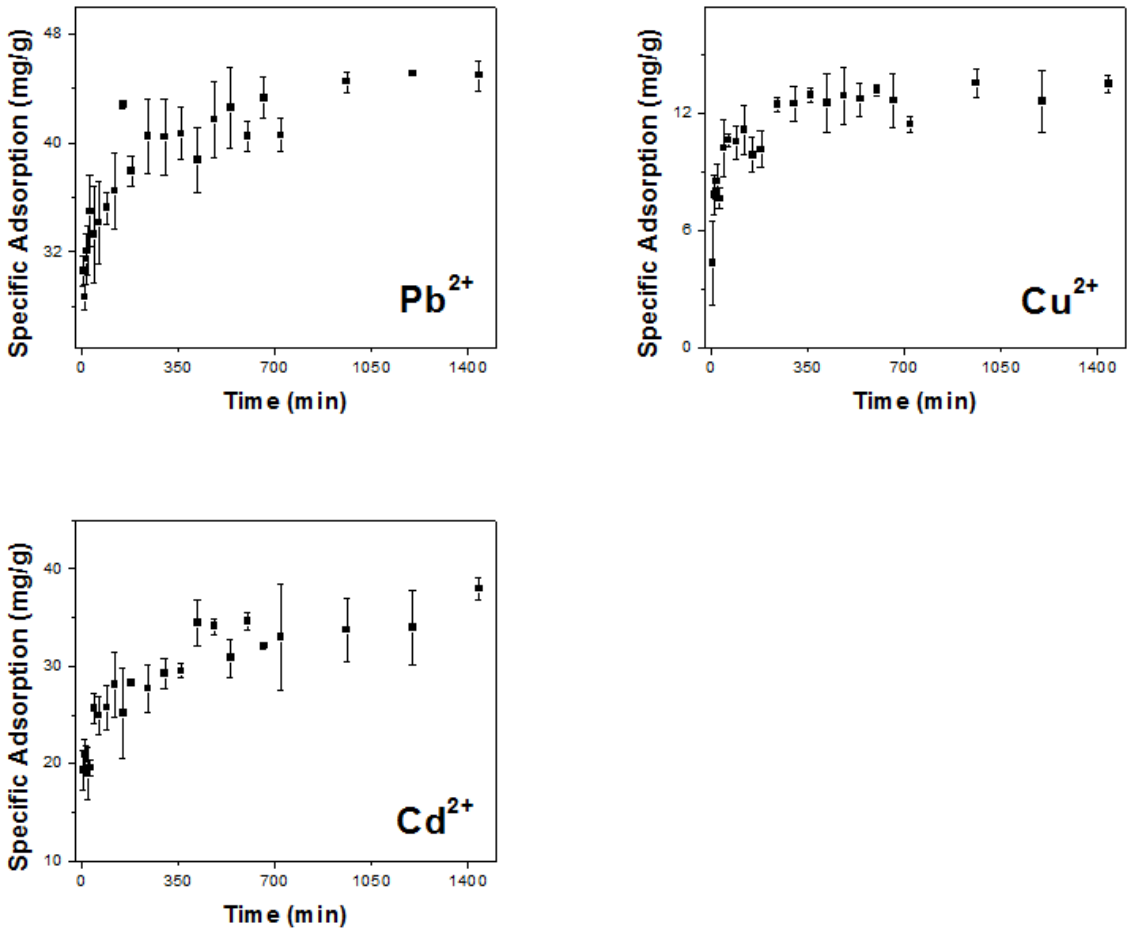

Figure 2.7 Kinetics for PSAC adsorption of lead, copper, and cadmium

The uptake of Pb^{2+} versus time was fitted to both pseudo first and second order kinetic models. The first order model is given as the following equation:

$$log(q_e - q_t) = logq_e - \frac{k_1 t}{2.303} \tag{2.1}$$

qe is the amount of metal adsorbed at equilibrium, qt is the amount of metal adsorbed at the specified time (t), and k_1 is the rate constant for first order adsorption. The second order model is given as the following equation:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{2.2}$$

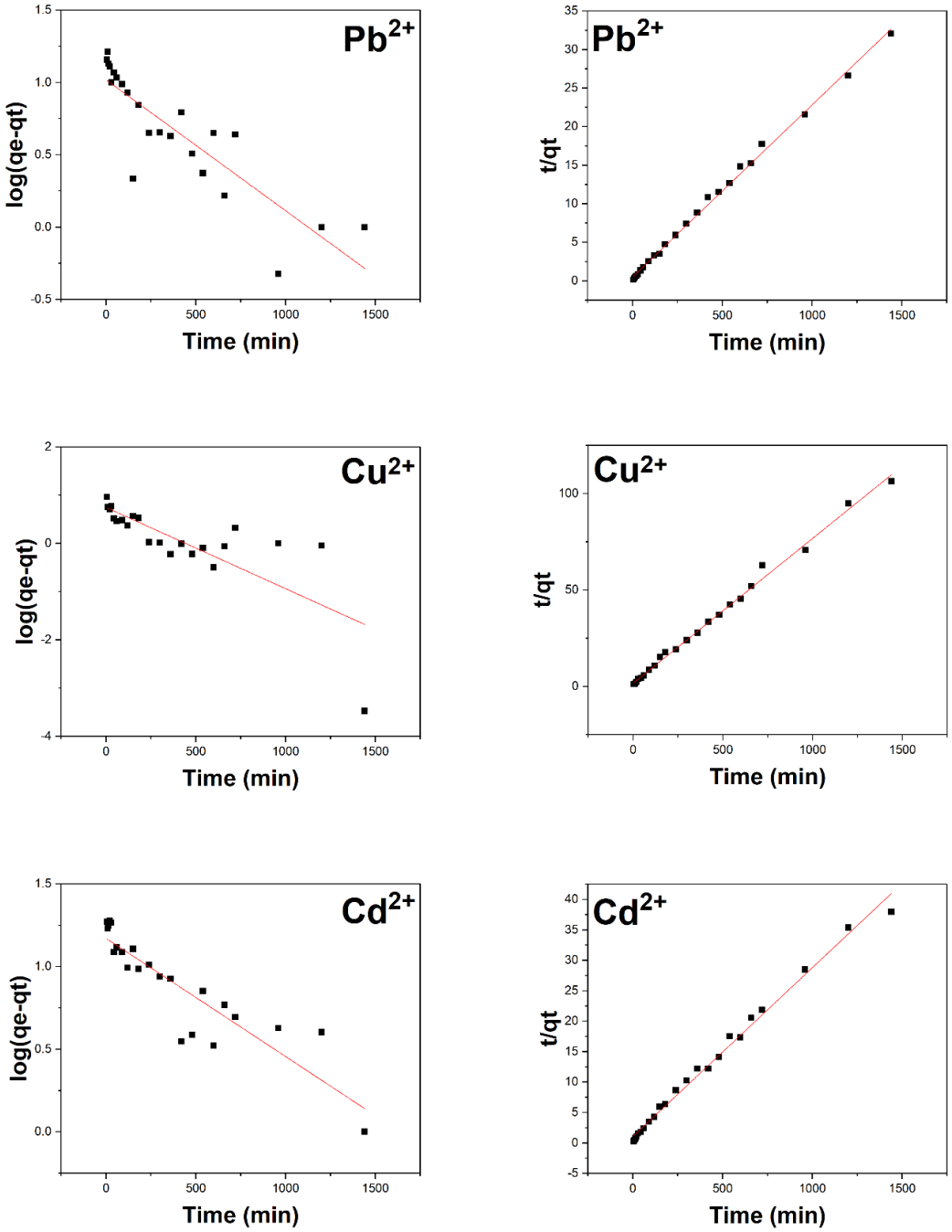

Figure 2.8 Linearized first and second order kinetic plots for each metal adsorbed

Figure 2.8 shows the first and second order kinetics linearized plots for the various metal adsorptions onto PSAC. $\log(q_e-q_t)$ vs t is the linearized form for first order, and t/q_t vs t for second order. The first and second order parameters for the adsorption of each metal onto PSAC can be found in Tables 2.4 and 2.5 below. The R^2 values calculated show that the adsorption is second order for each metal's adsorption onto PSAC.

Table 2.4 First order parameters for PSAC

Metal	**Initial conc. (ppm)**	**Q exp. (mg/g)**	**R^2**
Pb^{2+}	100	45.0	0.742
Cu^{2+}	100	13.5	0.581
Cd^{2+}	100	37.9	0.805

Table 2.5 Second order parameters for PSAC

Metal	**Initial conc. (ppm)**	**Q exp. (mg/g)**	**R^2**
Pb^{2+}	100	45.0	0.997
Cu^{2+}	100	13.5	0.995
Cd^{2+}	100	37.9	0.992

2.8 Conclusions

This study focused on the removal of lead, cadmium, and copper from aqueous solutions using activated carbon manufactured from peanut shells. The activated carbon was created through chemical activation using H_3PO_4 as a 1:1 w/w pre-treatment before being heated for 1 h at 450 °C, and then cooled under a nitrogen environment. The char was characterized using BET, TGA, PZC, and SEM.

Its optimum adsorption parameters were found by several tests. By measuring the adsorption at a range of pH levels, the maximum adsorption was found to occur at a pH of 5. The adsorption equilibrium times of 17 h for Pb^{2+} and Cu^{2+}, and roughly 24 h for Cd^{2+} were found by analyzing the kinetics over a period of 24 h, and that data fit a pseudo second order model for all metals. The maximum adsorption capacities for PSAC at three different temperatures, 25 °C, 35 °C, and 45 °C, were found by performing isotherm studies on the biochar and fitting the data to Langmuir and Freundlich models. The maximum adsorption capacities for each temperature were found to be 100.2, 87.8, and 84.9 mg/g respectively. Single layer adsorption of lead was indicated by the Langmuir model fitting with an R^2 value of 0.977 at 25 °C.

SEM imaging showed the surface of the char, before and after adsorption of lead and BET analysis gave the total surface area of 781 $m^2 g^{-1}$. The PZC for PSAC was 2.05, which means that the surface of the char was negatively charged when the pH of 5 was used for the adsorption experiments, making it a good adsorbent for metal cation adsorption.

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CHAPTER III

CHEMICAL EDUCATION AND THE IMPORTANCE OF A HANDS-ON APPROACH TO TEACHING

3.1 Introduction to Chemical Education

Chemistry is a topic that can be difficult to understand, one that is complex and takes work both to learn and to teach effectively. At its core, chemical education research is focused on enhancing chemistry learning, improving on teaching methods, and making it more accessible to those who struggle in the area[1,2]. There are many smaller goals in the field of chemical education research that can be examined, and they all feed into the overarching goal stated by Bunce and Cole (2008), of "how to help students learn chemistry more effectively and efficiently"[3]. This dissertation focuses upon two projects in chemical education that highlights student learning through the theoretical framework of constructivism.

Because of this desire to help students learn chemistry more efficiently, chemical education is a vast field. It encompasses all branches of chemistry, and the American Chemical Society has published a Science Education Policy declaring three specific objectives pertaining to chemical education[4]:

1) Promote lifelong, rigorous education of science concepts and practices in formal and informal settings to improve citizens' understanding of science and its role in society.

2) Provide adequate state and federal support for science education, as well as pre- and in- service teacher preparation and continuing education, to strengthen the quality of teaching which will enhance student learning.

3) Encourage students of all backgrounds, particularly those from underrepresented groups, in the pursuit of education and careers in STEM fields.

These objectives outline the main focus of chemical education as a whole, by promoting lifelong STEM education as a service to society, supporting and preparing teachers to enhance the quality of STEM education, and increasing STEM engagement for all students with a special focus on those that are underrepresented.

3.2 Approaches to Chemical Education

Among the many approaches to chemistry education, there is a theme that a lot of the more creative approaches have in common, that of a hands-on approach to learning. Instead of pushing for the students to spend more time listening to lectures, the instructors are providing them with alternative exposure to the materials already presented in lecture, allowing for alternate methods of learning.

Much research has been done on active learning as a whole, to determine the effectiveness of this approach that is so different from teacher-centered learning. Michael[5] has summarized findings from a variety of fields, and has described the 5 key findings that must be considered when discussing science education.

The first key finding summarized by Michael[5] concerns the fundamental tenant of constructivism, which is defined by Driver et al. as "The view that knowledge cannot be transmitted but must be constructed by the mental activity of learners underpins contemporary perspectives on science education"[6]. This idea centers around the combining of old information with newly acquired information, facilitating learning through the process.

The second key finding is recognizing that there is a difference between learning information and learning how to do things. To make sure that students learn the ability to do things, they must be given the opportunity to practice the skills being taught to them and given feedback in order to improve and retain that knowledge. The third key finding is the concept of transfer, and how the

learning of one topic may affect the learning of another. This can be either positive or negative, but instructors and researchers should be aware of this process and understand how students may or may not be transferring information from previous experiences.[5]

The fourth key finding is simply that students generally learn more when they learn with other people, rather than learning alone. The fifth key finding is that students learn better by discussing their learnings to either their peers or mentors.[5] We saw this within our REU program, where the students gave short "elevator-pitch" presentations of their work which helped them to familiarize themselves with the project, as well as communicating it with other people to build their knowledge of the research language. We also saw this in the lab experiment photo diary aspect, where students would upload their experiments and use the diary to show what they were learning and experiencing.

There are many approaches to using active learning in the classroom to bolster student engagement. They range from structuring homework around virtual laboratories[7], using a "Jeopardy" like game in an inorganic classroom[8], performing a chorus and opera to herald global warming[8], all the way to introducing art activities to Ph.D. candidates that use visualizations to alert them to potential gaps in their knowledge[8].

Lin et al.[9] performed a study using 8th grade students and their chemistry teachers, in which the students were given a Student Conceptual Understanding Test (SCUT) on stoichiometry. The teachers chose the time that would be spent

teaching the concepts for the exam and were then asked to take a questionnaire in which they estimated their students' results. The results from this study showed a significant gap between the grades that the teachers estimated and actual student performance. This again brings to light just a few of the important themes of chemical education, in which the pacing of lectures, communication between students and both their peers and instructors, and having a realistic view of the efficacy of current teaching methods are all crucial topics[10].

According to Ling et al. (2020), "the beauty of chemistry is the motivation that attracts chemists to spend their lives exploring chemistry"[11]. As chemists, it is possible to invite new students to the field by sparking their interest through a multitude of ways. One such way is through hands-on learning, be that by augmented reality such as by Yang et al.[12] who used a chemistry learning app and paper blocks to simulate chemical reactions between elements to allow students better visualization of reaction. Hill and Nelson[13] developed an undergraduate safety course spanning all four years of the degree, where students could be more engaged in safety training as it went alongside with their coursework. Siodlak[14] circumvented the high cost of model kits by designing models out of plastic bottle caps, which could be assembled by students at home. These activities get students involved and interested in the subject they're being taught, as with the polymer semiconductor education kits that Enlow et al.[15] employed, which were met with great enthusiasm by both students and instructors.

3.2.1 Hands-on Instruction

There are several popular approaches to hands-on laboratory instruction, of varying levels of use across the field of chemistry education. The first is structured inquiry, sometimes referred to as "cookbook" chemistry[16], in which students follow a pre-set process to find the answers to questions set by the instructor[17]. One of the advantages to this approach is a consistent "path" of learning that every student will access the same way, but similarly could be a disadvantage as students tend to learn differently and some could learn better with different approaches if given the chance.

Another approach is process-oriented, guided-inquiry learning (POGIL), which employs learning cycles that utilize teamwork and engagement with the newly learned material in order to solve problems[18,19]. Advantages to POGIL are group learning and student-driven experimentation, and among the disadvantages is a need for students to be more self-driven in order for them to receive the most out of this approach.

The third major approach is cooperative learning, where students are put into small groups to work together on structured tasks set by their instructor[20,21]. Group learning, as we have discussed, is generally seen as an advantage as it promotes students to discuss their learnings with their peers and mentors.

Lastly, is the implementation of course-based undergraduate research experiences, or CUREs, in which neither the student nor instructor know the outcome of the research being performed, and the student approaches their research experience by utilizing skills learned in a specific chemistry course[22,23].

The advantages to this approach are a more “real-world” research experience for the students, which could interest them in further research. Though a disadvantage may be in a project not working out as hoped, that is still a reality of actual scientific research and could still be counted as a learning experience.

3.2.1.1 Advantages to Hands-on Instruction

These hands-on teaching methods offer different avenues to increased student engagement, such as Finkenstaedt-Quinn et al.[24] and their use of TED-Ed videos paired with hands-on chemistry demonstrations. Students instructed utilizing this pairing exhibited increased understanding and confidence on the topics covered, all while being provided knowledge on relevant real-world topics and their relationship with chemistry concepts.

Jones[25] reported that students who prepared for a lab by completing multimedia lessons scored significantly higher than students who prepared for the same lab by reading. They also compared paper-based assessments of stereochemistry to visual assessments via computer, and the technology-based assessments were found to be similarly effective. Furlan[26] found that by allowing students hands-on experience with a scanning tunnelling microscope, engagement was increased and 95% of the students responded that the experience helped improve their chemistry attitudes. The students also stated that the research and lab experiences motivated them to learn more about chemistry.

Baum et al.[27] provided opportunities for environmental chemistry research to students who otherwise wouldn't have had the chance to experience research, and found through feedback that the research projects positively affected their future career plans. The feedback received also indicated that the students gained confidence in their abilities and competence with laboratory procedures.

Since individual teaching methods each tend to produce their own largely unpredictable results, researching these techniques provides a toolbox for those in this field to address the unique needs of the varying student populations.

Amidst the discussion of the hands-on approach leading to increased STEM engagement and skill building, the rate of STEM attrition in undergraduate education cannot be overlooked. Around half of students who declare STEM majors don't stay in their declared major throughout their undergraduate education[28], and among those that do stay, minorities are noticeably underrepresented as only 18% of STEM degrees conferred[29]. For those who do stay in STEM, it has been reported that many students are obtaining degrees while still lacking certain skills that future workplaces are expecting them to utilize[30,31]. Specifically, soft skills such as communication and collaboration as well as having broad knowledge within their field and written skills.

One way that students can work on obtaining skills is through exposure to laboratory research outside of that which happens within course work. By working in research, especially that outside the academic course lab, students can develop better communication and collaboration skills with their fellow

researchers, as well as having ample opportunity to practice scientific writing skills. CUREs provide this on a smaller level, giving students the opportunity to utilize learned skills and research towards unknown outcomes as a part of their education[32–34]. A further action that can be taken to give students the opportunity to develop these skills participation in a Research Experience for Undergraduates program[27].

In our work, we focused on two hands-on approaches to chemistry education: the formation of a Research Experience for Undergraduates program and the development of a home-based laboratory experience for an introductory organic lecture course.

3.3 Research Experience for Undergraduates

Undergraduate research, namely research performed in an academic or industry setting where the student is actively participating in research experiments, is an invaluable experience for any student[35,36], but particularly valuable for those students who are looking to pursue a career in science, technology, engineering, or mathematics (STEM). Undergraduate research provides laboratory experience outside of the rigidly structured laboratory components of courses, giving students a chance to experience the development, formation, and execution of planned research.

3.3.1 Opportunities provided by REU programs

Research Experience for Undergraduates (REU) programs[37] provide many students with research opportunities that would not be available to them otherwise. For many undergraduate students that attend non-research universities, there is no opportunity for laboratory experience outside of the classroom, which may lead to higher STEM attrition rates as mentioned previously.

There are several ways that these programs support students to stay within their chosen STEM field through the experiences offered[38–40]. By providing students with a stipend, they can earn a wage while researching and becoming for familiar with their field. By obtaining more lab experience, students can become more confident in their scientific abilities and may develop a further interest in STEM career paths. These programs are also opportunities for students to build a community amongst their cohort, where they can feel like they belong and thrive. All of these things, along with innumerable more, can attribute to students staying with and finishing their chosen STEM degree.

REUs provide research opportunities not only to those students at Primarily Undergraduate Institutions[41] (PUIs), but also students at two-year colleges, and those historically black colleges and universities that are undergraduate-focused. In the US, these programs are often funded by the National Science Foundation (NSF), the National Institute of Health (NIH), and/or by the hosting university. There are also many similar programs (titled SURE or SURP programs) hosted by medical schools and funded by the Association of American Medical Colleges

(AAMC). By providing these important opportunities to underrepresented minorities, first-generation college students, and women, STEM engagement can be spread to a more diverse audience through the REUs reach.

As students that attend PUIs may not have the opportunity to perform research on par with those who attend research universities, REU programs provide a field where those students can gain exposure to research and a wide variety of skills. Even skills such as presenting work at conferences and poster creation is one often overlooked, but that is frequently not available to students at PUIs[42]. By giving students these opportunities through a summer research program, it lessens some of the disadvantages of not attending a research-focused university and gives students a more competitive edge when applying to graduate or professional programs.

3.3.2 Typical Setup

The typical setup of an REU program is to utilize the summer, generally from 8 to 10 weeks, where students from across the country and sometimes even internationally[43] visit the host university for the duration of the program. Students are chosen from the applicant pool by the PI(s) of the program, and other faculty are brought in to be research mentors to the participating REU students. The participating students take part in research through the summer, building their own skillsets and learning the methods of research with the eventual goal of contributing to peer-reviewed manuscripts and conference presentations.

3.3.3 Measuring Success

Many metrics can be measured as to whether an REU program is successful or not, and many successes have been reported, though a caveat must be stated that there is little way to have a control group in these studies, and the actual reasons behind the reported successes could be varied. Various evaluation tools are used for measuring the success of REU programs, the most common are self-reported skill confidence surveys. Having thorough pre- and post- surveys provide extensive data as to the change made in students' outlooks specifically due to the program.

Amongst the programs we will review, there are both those who focus on the outcome of the program and those who focus on the changes made in the participants during the program. First, we will look at those that report significant effects of the program. These reports provide evidence that their program may lead to positive effects within the participant population.

In the program described by Gonzales-Espada et al. [44], their students were asked to rank their plans to attend graduate school on a scale of 1 to 5. 10.5% of participants lowered their final score after the program, while 36.8% raised their final score. The participants' career plans were modified according to the research experiences that they had throughout the program.

A study[45] was performed on two similar groups of students, one group having completed an REU program and the second having applied but not having

been accepted. There was found no statistically significant difference between the groups' pursued degree types at that time, but that within 6 years after completing their baccalaureate degree there was a significantly higher number of PhDs and lower number of MSs amongst the REU students. This data suggests that completion of an REU program may attribute to a higher chance of completing a PhD.

Raman et al.[46] found statistically significant correlations between mentoring and the students' overall REU experience. The students who said that their mentors performed highly in the focused areas also reported that they had a very good to excellent experience.

Among the second group, these programs have reported their outcomes but not the changes between before and after. While measuring and reporting success, it must be stated that self-selection bias is often in place when recruiting students for REU programs. As programs wish to promote further research such as graduate programs or professional schools, they often recruit students that are already interested in those paths. This could lead to reporting high levels of successful outcomes that would have happened without the program's influence.

Bennie et al.[47] reported success in their program's goals of motivating students into graduate programs and preparing students for research. Stone et al.[48] stated that their program met their goal of encouraging students from underrepresented groups in STEM in hopes of retaining them in their field, and

reported that their survey data showed almost 1/3 of their students continued to graduate school.

Grimberg et al.[49] reports that one of the main objectives of their REU program was also to convince students to attend graduate school, and that over 60 percent of their participants entered either graduate or professional school afterwards. They also stated, however, that this might be expected because of how participants are recruited and selected, but between the pre- and post-surveys, there was an increase in the number of participants that expressed a desire to pursue these opportunities.

San Andrés et al.[50] was yet another group whose goal was to increase interest in graduate school and measured their success by the proportion of participants who apply for and enter graduate programs or those who are supported for research following their participation in the program. They state that 90% of their participants indicated a desire to pursue advanced degrees. This may be attributed to selection bias of choosing students who already had these plans before entering the program, or the students could have developed that desire during the program.

Nadelson et al.[51], MacDonald et al.[41], and Nile et al.[43] all report on goals of increasing their students' self-confidence in laboratory work, and discuss how their programs accomplished that goal. These three programs employed pre- and post-surveys to measure students' perceived confidence in the skills they gained throughout the program, and Nadelson at al.[51] also used interview questions

designed to target the students' perceptions of how the REU program influenced them. Further metrics that increase the rating of an REU program are the inclusion of underrepresented categories of STEM students and prioritizing students from PUIs.

3.3.4 Research Objectives

Our REU study discusses the development and implementation of the MSU Department of Chemistry's NSF-funded summer research experience for undergraduates program, as well as the analysis of the student-reported results from their 10-week research experience. Two of our main goals were for students to experience confidence growth for a pre-defined set of skills, as well as allowing students to construct their own knowledge through experimentation and supporting student confidence in the defined skills, and our pre- and exit surveys showed consistent self-reported confidence growth among the student cohort in those skill sets.

3.4 Laboratory Components in Chemistry Education

As not all students will have the chance to take part in REU programs, another component that is instrumental to their STEM education is the inclusion of laboratory components coupled with their science courses. A great amount of research has been done where these undergraduate labs are concerned. Winkelmann et al.[52] found that moving towards research-inspired lab models

increased students' self-efficacy in completing inquiry-based activities, and that research-inspired labs provided some of the same positives as research itself. Cacciatore et al.[53] found that, upon adding research-based inquiry labs, the students in the new labs performed significantly better than the control group on experimental design and data analysis problems, but did not perform better on questions about general laboratory knowledge. They state that the findings indicate a positive treatment effect on student development of reasoning skills needed for experimental work.

Gao et al.[54] reports that enhanced learning was observed in introductory chemistry laboratories when using a inquiry-based approach, and that concept/skill training, feedback, and student-centered activities were supported throughout the semester. Cartrette et al.[55] designed a two-year laboratory course that focused on hands-on experiences with chemical instrumentation, as an instructional model for preparing students for work in a research laboratory. The course includes a stand-alone segment wherein the student designs and implements experiments on their own. They reported that nearly half of the participants went on to take part in some form of research activity afterwards.

Warner et al.[56] looked into the relationship between instrumentation use in curriculum and student learning. Not only did they find that emphasis on instrumentation in lecture appeared to increase perceived familiarity, but also that instrument use in the lab leads to increased technical knowledge in students.

They found that introducing new instruments to students, along with a guided inquiry-approach to labs, seemed to improve their problem-solving skills.

Damkaci et al.[57] designed and implemented a peer mentor program for undergraduate chemistry students, designed to improve STEM retention and work alongside of the general chemistry lab. The first four groups of students that took part in the peer mentored laboratories demonstrated a four-year average improvement of 20% versus those not in peer mentored laboratories, and 85.4% of the peer mentored students recommended continuation of the program. Fung et al.[58] also redesigned their senior-level undergraduate practical labs in order to give the students more experience to prepare them for research after finishing their degree, by utilizing Kolb's experiential learning cycle.

Galloway et al.[59] developed The Meaningful Learning in the Laboratory Instrument to measure students' expectations and experiences involved with learning in chemistry labs. They found that even when students had high expectations in general chemistry that went unfulfilled, their expectations entering into organic chemistry stayed high, and that the unfulfilled expectations didn't necessarily lead to negative experiences throughout the rest of their chemistry career.

Alongside much literature that supports labs as part of student success, there are also those who believe that the efficacy of labs for non-majors should be questioned. Hart[60] proposes that it is the presence of faculty that make the difference in students' performance, not the lab itself. Son[61] examined the

different impacts of in-person, virtual, and hybrid labs, and found that online labs resulted in higher grades than physical labs. When discussing control trials with students however, DeFeo et al.[62] states the difficulty of performing randomized control trials with students, alongside of the fact that these studies are usually performed by the faculty themselves who can't control all of the variables needed to make the study blind.

Despite these counterpoints, laboratory courses are still a useful and, in our opinion, necessary component of STEM courses. They provide students with opportunities to learn skills that they may not receive by other means.

3.4.1 Measuring Success

As the course our work focuses on was aimed at non-chemistry major STEM students, the definition of a successful laboratory experience for those students is varied, but many metrics are undisputedly part of the student success equation. Increased STEM retention[63], students relating chemistry concepts to real-world applications[64], improved academic performance[65], and increased interest in research participation[55,66] are just some of the many reported successes attributed to research being performed to improve undergraduate laboratory experiences. In 2016, the White House reported that by 2022 there would be a workforce need of 1 million additional STEM graduates[67], and an increase in any, or better yet, all of the above metrics, would vastly increase our chances of meeting that goal.

We measured the success of our laboratory experience through students' grades, self-reported surveys, and feedback from those who took the course.

3.4.2 Distance Learning and Online Instruction

In an age of ever-increasing internet usage and digital media consumption, the percentage of students taking online classes has risen dramatically in the last decade[68], and with the rise of the global pandemic COVID-19, a reported 97% of current college students have moved to online instruction[69]. Although distance learning has many advantages, such as completing degrees with a flexible schedule and from the safety of home, as of 2019 there was no fully online chemistry degree program. C&EN reported in 2019 on the first ever online biochemistry degree program, and noted that the university started with biochemistry instead of chemistry "because fewer classes in the biochemistry major have laboratory requirements"[70].

COVID-19 has brought this topic more prominently into focus, as many universities struggled to find ways to offer their chemistry labs online for the remainder of the Spring 2020 semester, as well as starting the Fall 2020 semester. They resorted to a variety of solutions. Some dropped physical experiments entirely for alternatives such as a virtual symposium focused on literature and writing[71], meeting via Zoom for laboratory periods in which a video of the experiment was played so the students could follow along and answer

questions[72,73], and one university live-streamed the experiments so students could observe and discuss the experiments in real time[74].

3.4.2.1 Home-based Lab Kits

One underutilized approach to chemistry distance learning is the development and use of kits containing the materials necessary for students to conduct home-based laboratory experiments. If a fully online chemistry degree that also upholds the importance of laboratory skills is ever to be offered, this is one way in which all the requirements could be met. COVID-19 led to quick development of some novel at-home experiments, such as a physical chemistry laboratory experiment using household materials and a smartphone to double as a photometer[75], a course of food-based kitchen laboratory experiments for nonmajors[76], as well as the introduction of a few small kitchen chemistry laboratory experiments using components most students would have at home[77,78].

Portland Community College offers a ¼ introductory chemistry and ¾ general organic biological course using student-purchased lab kits from LabPaq[79]. The kits have been customized for their courses and come equipped with everything the students need for the course. Peter Jeschofnig at Colorado Mountain College spoke of his work on developing kit-based experiments for first and second semester chemistry courses, as well as physics courses, as well as his interactions with other professionals that are developing or have developed micro-biology, organic chemistry, and geology kits[80].

3.4.2.2 Alternative Approaches

As of 2014 when this course was developed, no distance learning experience existed for the laboratory component of a survey of organic chemistry course. Kits existed for other courses, as mentioned in the previous section. Pre-COVID, there were a small variety of home-based lab kits for sale, mainly focused on general chemistry. They are made by companies such as Carolina[81], eScience Labs[82], Flinn Scientific[83], and Quality Science Labs[84], and are sold to the public, to be used with outside curriculum rather than a structured lecture-based course. Some universities offer alternative methods, such as kits for analytical laboratory experiments with instructions included on CD[85], kits for general chemistry laboratory experiments that lasted for the entire semester[86–88], general chemistry kitchen laboratory experiments that only use household items[89], and kits for micro-scaled general chemistry experiments[90].

These labs all provide the basis for further work into home-based "kitchen chemistry" kits for courses above general chemistry, where the only online option found for courses such as organic chemistry, was online laboratories such as Virtual ChemLab by Pearson[91], which is no longer offered.

Since 2020 there has been a rapid increase in the number of distance learning experiences available, to fill the need left by the pandemic. Kelley[92] developed a hands-on, at-home kit for organic laboratory activities aimed at high school students. Ibarra-Rivera et al.[93] designed an at-home column chromatography experiment for an Organic Technique Laboratory course. Schmidt et al.[94] created a set of organic and polymer chemistry experiments that

used household adhesives, that could be done at home by undergraduate students.

The University of Michigan-Dearborn offers an organic chemistry course with a fully online lab[95], where students can watch instructor-recorded experiments, simulations, demonstrations, and use an interactive textbook. They have worksheets and reports for student evaluation, as well as exams.

NC State offers a novel way for students to perform organic chemistry labs via distance learning – through virtual reality[96,97]. Their program covers the entire first semester of organic chemistry, and their results show no significant difference in the performance of students that did a traditional lab vs the VR experience. These results suggest an effective alternative for distance learning of organic chemistry.

3.4.3 Research Objectives

Our work focused on the development, application, and analysis of results for a novel home-based laboratory component of a semester-long survey of organic chemistry course. It featured 12 lab activities: 8 hands-on experiments and 4 online modeling exercises. By developing and sharing this off-campus approach, we hope to provide an option for other universities that are looking for at-home laboratory experiences for their own students.

Our program was evaluated through examination of student worksheet grades and self-reported student surveys. The students were asked questions

such as if the labs helped them to understand their course, as well as questions relating to the experiments themselves. This data was analyzed to determine the efficacy of the lab course. The results from our REU program will be discussed in chapter 4, and the results from our home-based survey of organic chemistry lab course component will be discussed in chapter 5.

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CHAPTER IV

REU-INFEWS program: Results from Innovations at the Nexus of Food, Energy and Water Security Program

4.1 Abstract

From 2017-2021, Mississippi State University's Department of Chemistry has hosted a 10-week summer Research Experience for Undergraduates (REU) program, focused on research related to food, energy, and water security topics. The goals of this program were to train students by providing an intensive research experience, recruit minority and underrepresented students, and provide advising for future career goals. The program hosted a growing cohort of undergraduate students each summer, 113 in total, recruited from a pool of underrepresented students and those with limited research opportunities. The pre- and post-program survey results covering three summers showed consistent self-reported growth among the student cohort in the program's focused skill sets. This manuscript presents three years of the program's success, from initial planning stages and recruitment to final results including a description of the value of each program component.

4.2 Introduction

Undergraduate STEM students can be encouraged to remain in STEM fields through research-focused experiences that allow better access to research-involved faculty [1-3], more modern or needed facilities [4,5], or a research-focused curriculum [6-10]. A number of summer programs exist to expand these opportunities to students at smaller Primarily Undergraduate Institutions (PUIs), community colleges and liberal arts universities [11]. Students in programs at smaller institutions may graduate never having performed research or experimental work outside of the rigidly structured curriculum-coupled laboratory courses. Exposure to external research experiences is particularly beneficial to these students and can support continued student engagement with STEM.

Our program promotes inclusion of underrepresented students in STEM fields to address an increasing national concern. Students of color face barriers in education, especially within STEM education, which lead to much lower proficiency in STEM courses in K-12 which negatively effects their university readiness and performance [12]. Women face more internal and external barriers than men, such as sexism amongst the male-predominant STEM environments, as well as cultural barriers such as pressures to have children [13] and expectations of being more family-oriented instead of career/academic oriented. And if those barriers are successfully navigated, there are still the economic barriers previously mentioned in the fact that research opportunities are harder to find

for minority students and may be turned down because of financial need and a necessity for the student to work instead [14].

When students are able to access authentic research experiences, it is believed to promote a higher interest in furthering their education [15] as well as teaching students a multitude of skills that they may not have had the chance to acquire through traditional laboratory courses [16]. Allowing students to integrate their own ideas into their practical chemistry work gives them a feeling of responsibility and control over their learning [17], makes them more likely to enjoy and succeed in science [18], and allows them to "be involved in the exploration of something new" [19].

The summer-based Research Experiences for Undergraduates (REU) programs are funded through the National Science Foundation, with over 700 opportunities available throughout the United States. REU programs are an important supplement to the undergraduate student degree experience, and are believed to promote higher academic success in STEM courses [20,21], increase retention in STEM degrees [22], and support higher inclusion of minority students[23,24] in STEM fields.

A typical REU program supports a ten-week research experience over the summer, and provides students the opportunity to participate in individualized, hands-on research, working under both graduate student and faculty mentors. These programs provide many students with the opportunity to experience research earlier in their academic career[25], as well as giving some students access

to training and experiences unavailable at their home institution. By working with knowledgeable researchers, students who might have not had the opportunity to perform hands-on research outside of classroom environments can receive invaluable experience by guided scientific discovery.

Recruiting a diverse cohort of students is the groundwork to REU programs, and there is much importance in choosing both students who are qualified and those who the experience would provide opportunities that they would be unlikely to experience otherwise [26]. As our program focuses on recruiting underrepresented populations, we believe we have succeeded in providing many students with research experiences that were otherwise out of reach.

A main goal of all REU programs is to encourage and motivate students towards pursuing further STEM research work and graduate careers [27]. By providing our participants with a full summer of both academic and social experiences, as well as giving them access to the wealth of knowledge provided by research advisors and graduate student mentors, our program has followed in the footsteps of those before us in encouraging and promoting graduate school and STEM professional fields as possible career trajectories for our cohort.

My role within this project was to support the PI and Co-PI by taking part in planning sessions, brainstorming ideas for student engagement within the 10 weeks they were at our university, was a graduate student mentor to REU students each summer, as well as helping lead the cohort's weekly activities when the PI

and Co-PI were away on their study abroad. I also performed the data analysis and organization of materials.

4.3 Overview of Program

The aim of this Mississippi State University Department of Chemistry REU program (now in its 5th year) was to expose undergraduate students to research experiences regarding interrelated Food, Energy, and Water Security issues (Innovations at the Nexus of Food, Energy and Water Security; REU-INFEWS). We have offered a productive training ground for our country's future leaders in areas of renewable energy, water purification and the science of soil amendment to enhance food security. Targeted students for this program included undergraduates in the majors of chemistry, biochemistry, engineering, and other related STEM fields. We focused on recruiting minority, first generation and women students into our program and our recruiting efforts were aimed at the rich student pool from undergraduate institutions, HBCUs, and community college programs located in the Southeastern United States that typically have limited research opportunities. Our program exposed undergraduate students to a breadth of research on related Food, Energy and Water Security topics and allowed for connections across disciplinary boundaries.

Figure 4.1 Participating MSU NSF INFEWS REU students and mentors.

Also shown are 29 students participating in MURPs (Mlsna Undergraduate Research Program) that also enjoyed a summer research experience. Group photo (research students and mentors) taken at the Shackouls Honors College Undergraduate Research Symposium 2019.

This program hosted 8 students each summer (2017-2019) and 10 students 2020 (funded through 2024) through the NSF-REU program. Additional students were included in the research experiences, summer training workshops and social activities as an expanded research cohort (Figure 4.1). Some students were paid from external individual professor funded programs, some from independent scholarship programs, and some students were from our host institution and financially supported through the Chemistry department. Additional students also included were doing research for course credit during the summer. This has brought our total cohort of students to approximately 38 per summer (2017: 32, 2018: 39, 2019: 42). An important partner for us has been the Jackson Heart Scholarship Program which financially supports students from Tougaloo College, an HBCU located in Jackson, Mississippi. This program,

combined with financial support from our MSU Provost office, has funded 9 students to participate in the summer research experience. Many of our summer students were hosted in the MSU dorms which created a vibrant support community for student socialization. In addition, the inclusion of local MSU students expanded the social connections for out-of-town participants.

The program was housed within the Department of Chemistry at Mississippi State University (MSU) and included professor mentors and research groups from four colleges and seven departments at the university, including Chemistry (College of Arts and Sciences), Biochemistry, Landscape Architecture and Plant and Soil Sciences (College of Agriculture and Life Sciences), Civil Engineering and Biological Engineering (College of Engineering) and Sustainable Bioproducts (College of Forestry). Our ten-week summer program included seminars on career paths and entrepreneurship with a focus toward small business and regulatory concerns in environmental industries. Summer housing, instrumentation and laboratory access have been provided through Mississippi State University.

4.3.1 Program Research Focus

Mississippi State University has a rich history of interdisciplinary research aimed at the utilization of biomass[28-35] as our warm weather and plentiful water allows for long and productive growing seasons. Our ability to produce high-density, high-volume biomass within the region is the foundation of this

bioproducts program and overlaps with a targeted research focus area of our institution. Many research groups at MSU actively conduct research aimed at converting biomass and microbes into renewable sources of energy[36–39]. During the production of biofuels we also produce biochar, a 'waste' product which can be used to purify water and amend soil depleted from years of intensive agricultural utilization[33,40–43].

Mississippi is a state with strong agricultural and forestry industries and the core of this program is the combination of research training for students entering environmental fields with an understanding of regulatory concerns, environmental fate and hazards of contaminants, and engineering approaches to support practical solutions to agriculture, energy and water environmental issues. In addition, our program has a focus toward the start-up and operation of a small business, so that students can understand pathways forward toward launching and sustaining an entrepreneurial effort.

4.3.1.1 Objectives of our REU site

1. Train students on state-of-the-art instrumentation and provide intensive research experience within the breadth of Food, Energy and Water Security. Projects emphasize environmental issues, renewable energy concerns, water purification and soil amendment.

2. Our program recruited talented minority, first generation, and female students from institutions with limited research opportunities. We have included students from all over the United States with strong emphasis toward the Southeastern region.

3. Seminars complemented the skills and research training with career advising, training in scientific communication, and identification of career goals and trajectories. Students practiced discussing their projects to outside groups and presented their work at an Undergraduate focused Research Conference at end of the 10-week program.

4.3.1.2 Overarching Goal

The overarching goal of this program was to support under-represented groups toward careers in STEM fields, with an emphasis toward graduate school and advanced degrees. We have included additional students supported through external funding sources within each summer cohort so that career advising, and training is expanded to more students. In this paper, we share what we have learned with the recruitment process, student training and experience along with the outcomes of this program. Elements that have made our program successful will be discussed for possible inclusion into other, existing, and future programs with a STEM research training focus.

4.4 Program Design and Evaluation

4.4.1 Recruitment and Selection of students

Our advertising materials for the REU-INFEWS program was shared via email with science department heads and undergraduate coordinators throughout the Southeastern United States. In addition, recruiting flyers were shared at National American Chemical Society conferences and the Southern Undergraduate Research Conferences to connect with students from Primarily Undergraduate Institutions (PUIs). The PI and Co-PI especially encouraged colleagues at HBCUs in the State of Mississippi and surrounding regions to promote a strong response from HBCU student applicants. As our program received recommendation letters for students, we retained the professor email addresses in a database to be included in subsequent year recruiting efforts. Thus, our number of applicants grew each year.

Our program was awarded initial funding in March of 2016. Our cohort from 2017 was selected from 70 applications, with significant growth in applicants in 2018 and 2019 (130 and 159 applications respectively). Each student applicant for the program was tasked to provide two letters of recommendation from math or science professors, we also required student essays on their previous research experience (if any) and their intended career goals.

4.4.2 Funding and Housing

The 10-week program as funded by the National Science Foundation provided a stipend of $500 per week, along with dormitory housing and a meal stipend. The dorms at Mississippi State University were initially assigned as single occupancy residence in the cheapest dorm option (2017, 2018). In 2019, administrators at MSU collaboratively agreed to allow REU summer research students to house as double occupancy in the premium dorms for a reduced housing rate. This housing switch worked extremely well for the students in summer 2019 and demonstrably increased student satisfaction as queried through exit interviews. These exit interviews provided an indication that our REU participants preferred having a roommate in the dorms (over having single occupancy) and vastly preferred a private bathroom over a communal hall bathroom. The INFEWS program collaborated extensively with other REU programs housed on campus to offer social connections and opportunities for student engagement. Access to the gym facilities was provided at extreme discount for the summer, along with parking passes and access to all university academic facilities. Participating students took advantage of these perks throughout the summer and helped to promote an enjoyable student experience.

4.4.3 Deadline Determination

Our initial application deadline was March 15th for a program that started at the end of May. We have adjusted our application deadline earlier (now March

1st) with an initial selection of students beginning in February. We have found that high-achieving students are in demand with all REU programs and are applying to multiple programs. Late communication with the student often means they have already committed elsewhere. We have therefore found that early communication in the selection process (early February) is crucial for recruitment success. Students are tasked to respond to the program invitation within one week if possible so that the next candidate can be selected in event of a rejection.

4.4.4 Application and Student Requirements

Our application is available on the Department of Chemistry website with questions on demographic background and math/science courses taken with grades. Three essays are requested: description of previous research experience, career goals, and why student is interested in this summer research program. A holistic approach was taken for applicant consideration and essays weighed heavily in the process with priority given to students expressing interest in research and career goals of research scientist. Two reference letters were part of the application process sent directly to program administrator and we prioritized students that demonstrated strong work ethic and the ability to work as part of a group. In addition, students were asked to rank available projects on their degree of interest. Project ranking often became a determining factor in selection as our program filled.

4.4.5 Selection Criteria

Priority for student selection were the following criteria: gender, ethnicity, first-generation criteria in addition to GPA, courses taken and letters of recommendation. Our program attracts students from a variety of majors, and we have included students majoring in engineering (chemical, civil, mechanical, biological, and environmental) along with students majoring in chemistry, biochemistry, biology, and environmental sciences. Students have been included after freshman, sophomore or junior years with some students matriculating from community college programs. Since many of our projects focus on the chemical sciences, most students have completed organic chemistry courses before program start although some students with less chemistry background have been included on projects that would be successful with less chemistry knowledge.

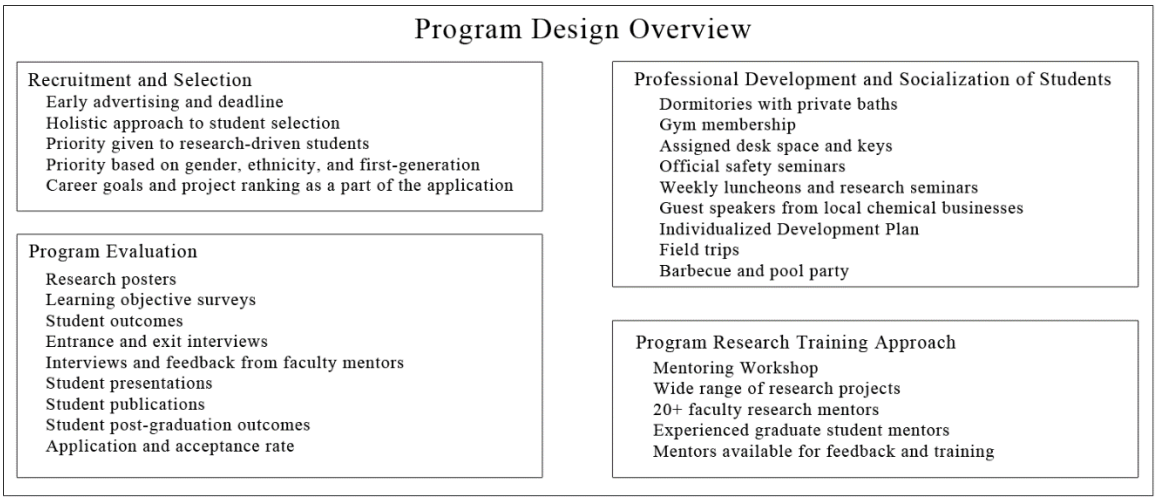

Figure 4.2 Program design overview.

4.5 Program Research Training Approach

4.5.1 Mentoring Workshop

Our program scheduled a faculty advisor and graduate student mentoring workshop 4 weeks before program start. This mentoring workshop had two primary goals: One was the outline and planning of a 10- week summer undergraduate research project that seemed achievable for the student. The project outline included specific goals and timelines with a recognition that our summer program is keeping students engaged and busy for 40 hours per week. We encouraged project timeline to revolve around data collection for a published manuscript, with identified goals that would result in publication of results. Project plan creation four weeks prior to program start allowed the research groups to consider chemical and supply needs with enough time to order anything that was missing. Each mentor was given up to $500 for student

supplies. The second goal of the mentoring meeting was to discuss interpersonal interactions and mentoring of undergraduate students. Discussions focused on communication of expectations, possible conflicts, methods to resolve conflicts and tools were provided to the graduate students to support their time management planning for the summer.

Selected REU student participants were matched with their research group before the program start, and their research project was included in their offer letter. Most of the research skill development was mentored by the faculty advisor and graduate students of that project group, although official training for library resources and SciFinder was included during our first week orientation (further explained under social components of program). Important deadlines for our program included an "elevator pitch" in week 2 that encouraged each undergraduate student to share the overview of their project and their defined goals for the summer research. Each student also presented a poster of their summer research at the end of summer Undergraduate Research Conference hosted by our institutions Honors College and turned in a draft manuscript report of their summer results to their faculty supervisor.

4.6 Research Projects

The overarching research theme of this REU program was on Food, Energy, and Water Security and each student was assigned their own individual research

project in areas relevant to this focus. The specific research areas included projects addressing issues with energy depletion, freshwater contamination, and food security. Over 20 faculty members participated in this program as research mentors, as well as multiple graduate students. Specific areas of research that were focused on include those listed in Table 4.1.

Table 4.1 Areas of research and their focus

Areas of Research/ Project
BioSyngas/ Bio-Oil
Development of multi-functional catalysts for conversion of cleaned syngas from biomass to hydrocarbon fuels
Upgrading of biomass fuels with plastic additives
Development of novel methods and catalysts for the production and upgrading of bio-oil
Biochars
Development of biochars for soil amendment and carbon sequestration
Development of biochars for water remediation
The impact of biochar on plant mycorrhizae and impact on plant growth
Exploring the relationship between the chemical mechanism of adsorption and the performance of various biochars
Developing biochar-based adsorbents to control sewage and agricultural runoff
Materials and methods for removing excess nutrients from agricultural runoff, thereby reducing eutrophication
Other
Using advanced NMR techniques to monitor the performance of green adsorbents with complex mixtures
Analysis of microplastics in marine animals using optical spectroscopic methods
Utilizing wastewater as a valuable source for water and energy recovery through microbial desalination
Development of bio-degradable polymeric materials

4.7 Professional Development and Socialization of students

Upon campus arrival, our students were checked in to their dormitory housing on campus. Our initial program utilized single-occupancy housing in our cheapest dormitory available with shared bathroom facilities on the hallway. In year 3, we were able to afford double-occupancy rooms in our expensive dorms where each student room had a private bath. The dorm rooms had a full-sized refrigerator and microwave in each room, plus communal kitchen facilities on the floor. Our students had access to all university facilities, including a campus gym membership for the summer ($2 for all summer access) and reduced rate parking ($40 for summer). Campus food courts and dining halls were available, and students could use their paid meal stipend to access restaurants or groceries. The first week of the program included an initial orientation to the program schedule and available amenities with an orientation packet that included local restaurants, activities and the campus bus schedule. The first morning of program was focused on this orientation overview followed by connecting each student in with their designated research group so they could meet group members, get assigned desk space and keys, and get oriented with research. The remainder of orientation included official seminars on laboratory safety, hazardous waste disposal, library orientation, SciFinder training and introduction to campus services and amenities.

4.7.1 Weekly Seminars

Two weekly seminars were scheduled for the ten weeks of summer. One scheduled every Wednesday at lunch with meal provided, focused on the Responsible Conduct of Research, Career trajectories in STEM fields, information on how to get in to graduate school, an Individualized Development Plan document (2 sessions), how to create a poster for a research conference presentation, and how to write a research manuscript. Our program secondary focus is on small business development and entrepreneurship, so several lunch seminars included guest speakers from local chemical businesses with tours of their facilities.

A second weekly seminar featured faculty speakers. These talks comprised a short overview of research focus (15 minutes), but a larger portion on their career trajectory, why they made major life choices during their career and what they wish they had known. Students were very engaged with these talks and have commented on them extensively in exit interviews. At this stage in their academic training, many students were eager to learn about the range of possible paths forward towards successful careers. The comments reflect that it helps them realize that there isn't just one "right" pathway.

Careers can happen from more than one defined pathway, and students have commented that it vastly reduced stress about their future to recognize that a variety of routes can lead to rewarding and productive careers. We discovered that our faculty really buy in to this discussion and enjoy the mentoring

interaction as a way of dispensing life advice to the students. The common theme was that faculty shared how they came to their current position. No two faculty stories were alike, and students gained the understanding that there are many paths that can lead to success. This series of presentations served provided an important life lesson for many of the students who began to understand that 'bumps in the road' towards a career were common and that persistence often was rewarded.

4.7.2 Individualized Development Plan

The Individualized Development Plan (IDP) was a paper document used with students to identify career goals and specific skills and experiences that would help them toward their defined goal. These documents allowed us to pursue conversations with the students on life priorities as well as the specific experiences the students could pursue to help expand their resume and grow their marketable skills. A specialized discussion with this IDP centered on the components of a strong recommendation letter, what the student identified that they would like said in a letter written about them, and specific approaches the student could do to garner those statements. For example, a student might identify that a strong statement in a rec letter is "strong work ethic". We would go through ideas and examples of how a student could manage their summer experience and their interaction with their research group to garner that statement in a future rec letter. A strong theme for the IDP discussions was that

the student had the means to identify specific skills or outcomes that could help them toward their future. We would then explore ways to access those skills or support the defined outcomes. Many students commented in our exit interviews that this process was the point at which "life got real". The targeted discussions helped support students to recognize that they can be active participants in their growth and can both identify and pursue their defined goals.

4.7.3 Socialization Opportunities

Participants were given many opportunities to connect with each other, such as field trips and other social events. GroupMe chat groups were created to organize social outings, trips to the gym, and weekend activities. The inclusion of PIs varied from summer to summer; some participant groups were happy to keep their PIs involved in the GroupMe chat while others created splinter groups that included only student participants. In addition to these groups, Photo of the Week contests were hosted, encouraging participants to submit photos each week with various themes (selfie with their research group, best group photo, best nature photo, etc.).

Field trips included the Teacher Education EPSCOR workshop hosted at Tulane University and the University of Alabama, and the ERDC government lab in Vicksburg MS (2017, 2018, and 2019, respectively). A second field trip was held each summer based on the participants' interest, and those have included the Huntsville Space Flight Center, the MSU Energy Institute, and a local Water

Park. An opening barbecue and pool party welcomed participants each summer, and small evening and weekend social activities such as ice cream and movie nights happened approximately once per week.

4.8 Program Evaluation

Program objectives were evaluated in multiple ways. Written data such as student applications, research posters and learning objectives survey were analyzed and student outcomes were tracked to identify career trajectory post-program. Exit interviews were performed with students each summer to gain feedback about the program, campus experience and student training elements. Interviews and feedback from faculty mentors were analyzed to determine way to strengthen the program. These metrics are further discussed in Section 4.9.

Each following year, the program included new elements in response to prior surveys resulting in an ever-improving summer package. For student participant outcomes, we tracked student presentations and publications related to their research. We also track where students go after graduation from their home institution to determine their path - graduate school or other opportunities pursued. Continued tracking was achieved through LinkedIn connections, Facebook interactions and direct email contact.

To evaluate recruitment metrics, we analyzed overall applications and acceptance rate, towards our goal of recruiting women, minority, and first-generation participants and students from institutions with limited research opportunities.

4.9 Program Outcomes

4.9.1 Recruitment Metrics

We enrolled 24 REU students during the first 3 summers of operation. The first year, 2017, one selected student decided she had made a mistake in enrolling within the program after the first week and she asked to leave. We were able to roll her funding over into year 2 and support 9 students in 2018. Of the 24 total students supported within the REU, 16 were women, 4 were Black or African American, and 11 were First Generation. Eighteen students were from primarily undergraduate institutions (PUIs) or institutions with limited research capabilities. Three students were from Historically Black Colleges and Universities (HBCUs). Undergraduate institutions represented included Mississippi College, Huntingdon College, Tougaloo College, Jacksonville State University, Stetson University, Spring Arbor University, Tuskegee University, Georgia Southern University, Alcorn State University, Edinboro University and Thomas Nelson Community College. The majority of students were Chemistry majors (11 of the 24) with Chemical Engineering, Biomedical Engineering, Civil engineering, Biochemistry and Biology also represented.

For the REU program, 87.5% of our participants fulfilled our recruiting focus of women, Black or African American or First-Generation students. Additional undergraduate researchers were supported through external funding grants supplied by our research mentor faculty. An additional 9 students from Tougaloo College, an HBCU located in Jackson, MS were supported through private scholarship and funds supplied from the MSU Provost office and were treated as full program participants even though their funding came from a different source. The expansion of our cohort to include additional externally funded students allowed us to support a strong and diverse cohort. As well, local Mississippi State University students were supported through the Department of Chemistry, the MSU Shackouls Honors College and external programs to support local student research participation. A full breakdown of students is shown in Table 4.2.

Table 4.2 Student demographics among the funded student cohort

		NSF: REU-INFEWS Funded			**Externally funded Students**			**MSU Students/ half time**		
		2017	2018	2019	2017	2018	2019	2017	2018	2019
Gender	Male	3	3	2	3	5	6	4	7	9
	Female	4	6	6	3	2	7	2	5	10
Ethnicity										
	White	5	6	4	3	4	6	5	10	16
	Hispanic/Latino	1	1	1	1					1
	Asian American			2			1		1	1
	African American	1	2	1	2	4	6	1	1	1
	Other									
Other Factors										
	First Generation College Students	4	2	6		4	6	2	2	1
	Students from Historically Black Colleges	1	1	1	2	3	5			
Total Cohort 2017[a]	19	7			6			6		
Total Cohort 2018[a]	28		9			7			12	
Total Cohort 2019[a]	40			8			13			19

[a]Additional students were doing research for course credit

4.9.2 Research Outcomes

Twenty-four REU participants successfully completed the program and presented their research work at the final Shackouls Honors College Undergraduate Research Symposium. We had 2 students win awards with these presentations and 3 students presented their work at external conferences including the American Chemical Society National Meetings[44,45] and the Annual Biomedical Research Conference for Minority Students[46], 1 student won an award at a National ACS meeting. Another student won a travel award from the REU student committee to present their work in Washington, DC. An additional 89 students from our externally funded programs presented their work and 1 won an award at the Southeastern Undergraduate Research Conference. 5 students presented their work at external conferences, and a further 2 students had plans to present their work at the National ACS meeting in Philadelphia in 2020 but these plans were cancelled due to covid-19.

Six manuscripts[47-52] have been published from the REU program participants. We continue to strongly encourage our research mentors to publish their work and more manuscripts are in preparation. An additional 19 publications[53-71] have occurred with the externally supported students.

Dr. Deb Mlsna was an invited speaker at the 2019 SouthEastern Regional Meeting of the American Chemical Society[72], the 2020 International Frontiers in Chemical Technology Conference[73], and the 2018 National ACS Meeting[74]. Dr. Todd Mlsna was an invited speaker at the 2020 International Frontiers in Chemical Technology Conference[73], and the 2018 National ACS Meeting[74].

4.9.3 Student Outcomes

Six of our REU students have entered graduate school programs, with an additional 7 matriculated into the workforce or professional school programs (medical, dental, pharmacy school). Seven students are still completing their undergraduate degree. Approximately the same number of external students have also moved on to graduate school programs, and we continue to monitor the students to determine where they move toward after graduation.

Table 4.3 Self-reported Student Outcomes

	Graduate School	Professional School/ Employment	Still completing BS degree	No outcome/ lost track.
REU Students	6	7	7	4
External Funded Students	6	6	9	5
MSU Students	5	8	16	8

The pre-and post-surveys shared with students targeted our learning objectives for the summer experience and tracked student attitudes toward their own scientific confidence on 9 questions. Students were asked to rank themselves on a 5-point Likert scale as “unknown” (level 1) up through “advanced”

(level 5). Students consistently showed improvement in their skill sets after the ten-week program with the strongest learning gains observed with designing a hypothesis and writing a manuscript. Full results are shown below in Figure 4.3.

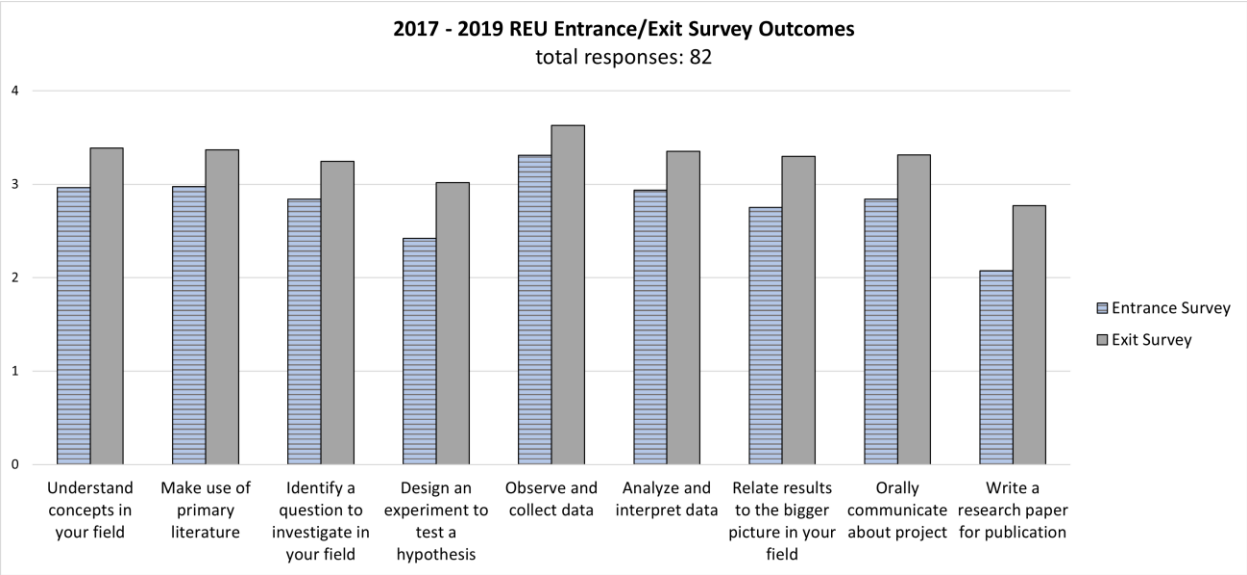

Figure 4.3 Learning Outcomes survey results.

With this evaluation, we hoped to address the question as to whether our participants were leaving our program feeling more capable at performing scientific tasks such as writing manuscripts and presenting their research. The evaluation surveys were designed to help students convey their confidence for these tasks with a simple number system and topics that specifically targeted areas of the program that we focused on. The data we received from these evaluations supports the conclusion that our program has indeed helped students feel more confident with these tasks.

4.10 Discussion

The learning objectives of oral communication and relating their project into the larger picture were supported through a collaborative relationship developed with an NSF EPSCOR-funded program, Creative Science through Inquiry (CSI). CSI was a teacher education workshop designed to support middle and high school science teachers to expand their skills in laboratory experiences. The REU participants visited this workshop each summer to interact with the teachers and support them to perform a biochar adsorbance experiment created by the PI and Co-PI to explain the programmatic research.

REU participants at this workshop were tasked to explain their summer research project in a 3-minute presentation for the secondary school educators. This early start presentation, given in the 2nd week of the program, yielded enormous value for our participants as it supported them to become familiarized with their topic and research focus very early in the summer program. The presentation consisted of two PowerPoint slides, the first giving detailed background and the importance of their summer research and the second detailing their project specifics including summer goals and a list of tasks. Scheduling an early student presentation was vital because it got the participants up to speed quickly with their project and fostered independence for the student in recognizing their own summer goals.

In addition, the interactions between the undergraduate REU participants and the teachers were brilliant to observe and was a remarkable team building experience. The undergraduates were able to step into a teaching role while the teachers got to see how much student growth occurs at the undergraduate level. Both teachers and students strongly endorsed this workshop experience for multiple years, and we believe that the completion of the CSI workshop helped both the teachers and students feel proud of their progress. As well, the learning objective of improving writing skills was supported through the student research poster at summers end and the submission of a manuscript draft on their project a program requirement. The reports were formatted to provide experience with writing a research publication, so all details such as introduction, methods, results, and discussion were required. These provided both invaluable scientific writing experience for the undergraduate participants, and a rough draft for their research mentors to expand on for future publication.

4.11 Conclusion

The authors feel that we have had a very successful launch to the chemistry NSF-REU program at Mississippi State University. We have introduced many underrepresented students to the benefits of STEM careers and helped to foster a further interest in pursuing these careers. All told, 113 students spent 10 weeks emersed in research while networking with potential colleagues in their cohort that may support their work for further decades. Along the way, they learned how

to prepare and give presentations, write research papers, maximize their resumes for graduate school, navigate research laboratories, discuss scientific principles with those in their field, and had countless other opportunities for progressing further into their chosen fields.

The program elements were supported through the three-year implementation of this REU-INFEWS program. The program continues at Mississippi State University through renewal funding and continues to expand in breadth and scope to support undergraduate students engaged in research. Successes with this program are the continued inclusion of additional participants supported through external funding programs and private scholarship. One of the most effective recruiting components was to include the faculty writing recommendation letters on the next year's recruiting list. The faculty that are engaged to support an undergraduate student with an REU application are excellent recruiters for the next layer of participants. This REU program has run successfully, has become a model program for our university, and continues to support the mission of engaging undergraduate students in authentic research experiences.

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CHAPTER V

DEVELOPMENT OF AN AT-HOME CHEMISTRY LAB EXPERIENCE FOR SURVEY OF ORGANIC CHEMISTRY STUDENTS

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5.1 Abstract

Educators at Mississippi State University developed an at-home, kitchen chemistry style laboratory to accompany the Survey of Organic Chemistry lecture course. The course featured twelve lab activities; eight hands-on experiences that focused primarily on important organic concepts including acid strength, extraction techniques, chromatographic separation, recrystallization and selected organic reactions. Four online modeling experiences used ChemSketch and three-dimensional visualization to support student understanding of stereochemistry, isomer configurations and important reaction mechanisms.

A unique component of this laboratory course was that students uploaded photo diaries of their experiments to demonstrate completion. The developed course was implemented and taught for three semesters at Mississippi State University with 119 students successfully completing the at-home, self-paced laboratory course. The authors share this at-home laboratory experience as an option for programs exploring novel, off-campus approaches to laboratory experiences.

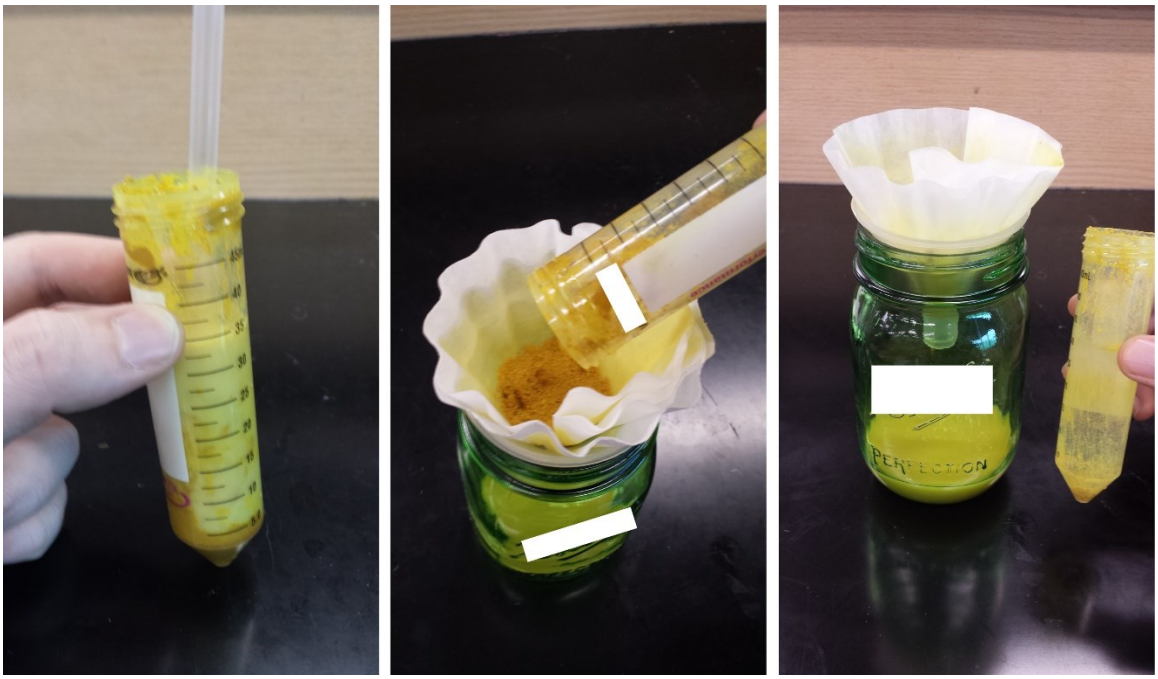


Figure 5.1 Graphical Abstract for Chapter V

5.2 Introduction

As internet access becomes more widespread throughout the United States, distance learning has risen in popularity. A wide variety of classes are offered in an online format, and many universities provide a broad list of degrees that can be obtained completely through online classes. According to Bay View Analytics,[1] the number of higher education students taking at least one online course has increased from 9.6% of students in 2002 to 29.7% of students in 2015. With recent university impacts related to COVID-19 closures, the number of students taking online coursework has now significantly increased. According to EducationData.org,[2] 97% of incoming undergraduate college freshman have switched to online classes as part or all of their college experience.

One area where distance learning may be lacking, however, is in laboratory-based chemistry courses. As these courses use hands-on lab experience to reinforce concepts taught in the classroom and prepare students for higher-level chemistry courses, the lab component is an essential experience. Several online general chemistry[3–7] and analytical chemistry[8] courses have been offered for distance learners, utilizing both an online lecture and home-based "kitchen chemistry" kits. Some companies have also began manufacturing chemistry kits[9–12] that can be purchased to perform experiments at home.

Our large public university previously had to offer their Survey of Organic Chemistry course through a distance learning format due to space constraints. The lecture course Survey of Organic Chemistry is designed for non-chemistry majors as an introduction to organic chemistry and requires at least one general

chemistry course as a pre-requisite. In support of the effort to expand this course to a distance learning, online format, instructors extensively sought an effective online experience that could support the laboratory curriculum.

Online laboratories such as Virtual ChemLab: Organic Synthesis and Qualitative Analysis published by Pearson were used as a distance lab component for approximately four years for this laboratory course, but the Virtual ChemLab program and software has been discontinued. In addition, while the Virtual ChemLab Organic Synthesis option was one of the few organic experiences available, there were difficulties using the program as the material does not overlap well with a Survey of Organic course. Limited online lab experiences exist for organic chemistry although some programs integrate videos and homework questions from an online homework site as a substitute.[13]

In response to the lack of offerings, this distance-learning, kitchen chemistry laboratory course was developed to elucidate chemical concepts emphasized in Survey of Organic lecture and support students to understand important organic laboratory techniques. The course ran successfully for three semesters at our institution (Summer 2014, Spring 2015, Summer 2015) before being transitioned over to traditional laboratory space that became available. This kitchen chemistry laboratory approach could be useful as programs explore online and at-home laboratory experiences for the Survey of Organic Chemistry concepts. My role in this project was the handling of all data entry, data analysis, organization of materials, and presentation of data within the manuscript.

5.3 Organization of Course

Our students were all registered at our university, so at start of semester, students came to campus to pick up a lab manual and box of supplies for the lab. The boxes students picked up contained Personal Protective equipment (gloves and safety goggles), laboratory equipment (vials, beakers, graduated cylinders and pipettes) and chemical supplies that were pre-mixed and pre-measured for use in an experiment. Additional supplies for the lab that students had to provide themselves included basic kitchen equipment (measuring cups and spoons, bowls, glasses, stove and refrigerator) and common grocery items such as salt and rubbing alcohol. Some items students needed to purchase (lemons, red cabbage) would have spoiled if pre-packed. In addition, students were also tasked to use their cell phone camera to construct a photo diary of their experiments. The online workbook assignments used ChemSketch as a free drawing and visualization tool.

Scheduled experiments for the course had due dates for each experiment and suggested timing that overlapped with lecture course material. Students had freedom and flexibility however to manage their own time and could complete assignments early if they chose different timing. Table 5.1 shows the list of experiments and the accompanying concept supported in the Survey of Organic lecture:

Table 5.1 Experiment list, Estimated Time for Completion, and Accompanying Concepts.

Experiment	Approximate Time Required, h	Lecture Concept(s)/ Laboratory Technique	Primary Learning Objective*
Recrystallization of Aspirin	24	Crystallization of organic compounds; removal of impurities	LO1
Acids & Bases using Red Cabbage pH Indicator	1-2	pH scale and pKa values; acid strength	LO1
Separation of Food Dyes by Chromatography	1-2	Polarity of compounds; chromatographic separation	LO1
Online ChemSketch Assignment: Molecular Modeling of Cyclohexane	2-3	3-D visualization of cyclohexane configurations; axial and equatorial positions	LO3
Extraction	24	Polarity; extraction techniques	LO1
Saturated & Unsaturated Oils	1-2	Saturated versus unsaturated compounds; addition reactions of alkenes	LO2
Online ChemSketch Assignment: Electrophilic Aromatic Substitution	2-3	EAS Reaction Mechanism; Substituent Directing effects	LO3
Online ChemSketch Assignment: Stereochemistry	2-3	3-D visualization of chiral carbons; identifying R and S Stereochemistry	LO3
Online ChemSketch Assignment: Alkyl Halides & S_N1/S_N2 Reactions	2-3	3-D visualization of stereochemistry and substitution reaction mechanisms	LO3
Making Polymers	1-2	Chain- and step-growth polymerization reactions	LO2
Preparation of Soap	2-3	Carboxylic Acid reactions (saponification)	LO2
Crossed Aldol Condensation	2-3	Aldol Condensation reactions, identifying acidic hydrogens	LO2

[a]Some experiments can support more than one learning objective. See the Identification of Learning Outcomes section for further discussion.

5.4 Safety Hazards

Students were required to submit a pre-lab, safety rules document prior to performing anything with an experiment. When doing these experiments at home, it was always required that the student wear the provided safety goggles. Nitrile gloves (provided) were also required for some labs. Students were cautioned to never eat or drink while performing an experiment and to wash their skin thoroughly if any chemical solution spilled on their skin. Each laboratory experiment contained detailed safety hazards in addition to appropriate waste disposal. Students were instructed to wash their hands with soap and water after performing each experiment.

5.5 Materials and Methods

Students were provided a pre-packaged box that contained PPE and specialized containers and chemicals for laboratory experiments. Since chemicals were included with the kit and we did not want to manage shipping costs, students were required to pick up the kit in person. In addition, the provided shopping list consisted of ingredients easily obtained at the grocery store or potentially already available in a typical kitchen. The items in the kit were paid for through student lab fees, but students were financially responsible for the "extra" grocery items. The list of items provided in kit and shopping list are shown below.

The checklist for the Survey of Organic at-home lab kit provided (one per student) includes these materials:

- Four 50-mL test tubes with caps
- Six 15-mL test tubes with caps
- Plastic funnel
- Two plastic 3-mL syringes
- Three plastic spatulas (look like straws)
- Four plastic pipets
- One 250-mL beaker
- Safety goggles
- Two pairs of nitrile gloves
- Lye solution in a 50-mL test tube
- PVA-based white glue in a 50-mL test tube
- Oil mixture in a plastic jar

These chemicals (per one student) are also provided in the kit:

- Oil A in a glass vial
- Oil B in a glass vial
- Oil C in a glass vial
- Cinnamaldehyde in a glass vial
- Acetone in a glass vial
- Iodine tincture solution in a dropper bottle
- 50% NaOH solution in a dropper bottle

- $KMnO_4$ solution in a dropper bottle
- Aspirin tablets in a resealable plastic bag
- Ground cloves in a resealable plastic bag
- Borax in a resealable plastic bag

Some equipment and related supplies that students need to do the at-home lab experiments are not included in the lab kit yet are likely available in their household, including these:

- Sink with hot and cold water
- Stove
- Refrigerator with freezer compartment
- Small cooking pot (to boil water)
- Bowl
- 4 clear glass jars with lids (jars in the range of 8–32 ounces or 236 mL–1 L will be fine); glass canning jars or empty, clean, reused preserves or condiment jars work well
- Measuring cup
- Butter knife, sharp knife, teaspoon, and tablespoon
- Small resealable plastic bags
- Paper towels
- Paper plate
- Rubber bands
- Ruler

- Paper clip or toothpick

Some items that students provide to do the at-home lab experiments are likely also available in their household, including these:

- Household chemicals such as vinegar, baking soda, soft drink, shampoo (see the Acid/Base experiment for more details; chemical list may vary depending on what is available)
- Ice
- Dishwashing or hand soap
- Table salt

Items that students must purchase to do the experiments (cost ~$10.00) include these:

- One 12-16 oz. bottle of 91% isopropyl alcohol (rubbing alcohol)
- Small bag of M&M candies or Skittle candies
- Food coloring (4 colors)
- Cornstarch
- One head of red cabbage
- One fresh lemon

5.6 Laboratory Process

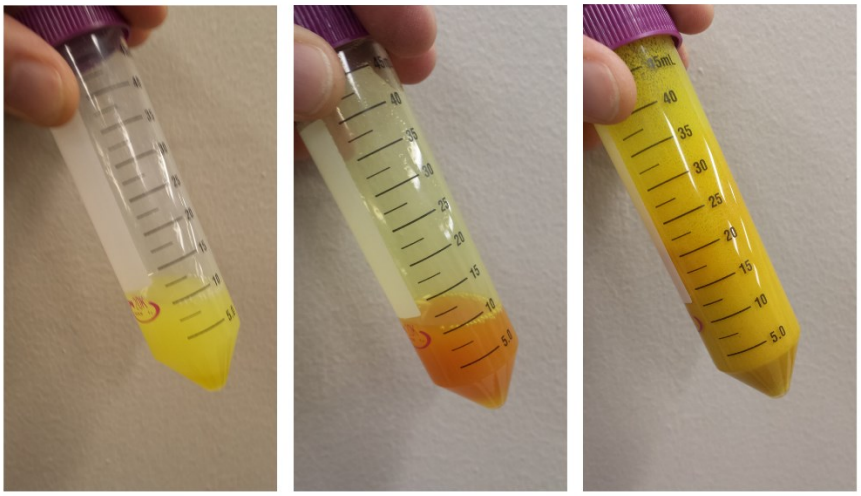
Students performing experiments at home had three required components to accomplish for each experiment. Each student had to complete a pre-lab exercise that was uploaded to the course with timestamp of upload recorded. Each pre-lab detailed safety information for the experiment and outlined the important learning objectives for students to observe. Students were cautioned that the pre-lab exercise must be uploaded before any experiment takes place. Second, students performed the experiment and took photos of the experiment as progress occurred. Third, the photos and answers to post-lab questions were then incorporated into a data sheet which was uploaded upon experiment completion.

Since we had just developed this laboratory experience, students also completed survey questions after every four experiments for us that provided feedback on the instructions and their success with the experiment. This allowed the instructors to connect in with student progress and provide more support if any confusion or safety concerns were noted. Figure 5.2 provides an example of the students' photo diary.

Experiment 12: Crossed Aldol Condensation

Photo Logbook **Name:**

1. Insert a few pictures of the reaction taking place over the 10 minute period.



2. Insert a picture of your final product after it has dried.

Figure 5.2 An example student photo logbook entry for the crossed aldol condensation experiment

5.7 Identification of Learning Outcomes

The learning objectives (LOs) for this laboratory are to:

1. Familiarize students with standard organic laboratory techniques (recrystallization, extraction, chromatography) in a hands-on environment
2. Support understanding of common organic reactions
3. Support student practice with stereochemistry and the three-dimensional visualization of organic molecules

5.7.1 LO1: Standard Laboratory Techniques

Each experiment was adapted from published protocols and modified for safe use at home. Standard laboratory techniques were supported through the following four experiments:

- Recrystallization of Aspirin[14,15]: Students recrystallized aspirin tablets using 91% isopropanol and water. The recrystallization process removes the binding agents included with aspirin tablets and produces purified acetylsalicylic acid crystals. Discussion of polarity and solvent choice is included to support student understanding of the purification of a compound through recrystallization.
- Acids and Bases using Red Cabbage pH Indicator[16,17]: Students purchased red cabbage and made their own pH indicator solution. Standard solution colors are provided as a visual key in the experiment, and students were

tasked to identify the pH of a variety of self-selected household chemicals. This experiment reviews acid/base definitions (Lewis, Bronsted-Lowry), the pH scale, and tasks students to identify relative acid strength, along with application of acid/base definitions to an example reaction mechanism.

- Separation of Food Dyes by Chromatography[18,19]: Coffee filters, purchased food dyes and colored candies are utilized to teach students about chromatographic separation of complex mixtures, polarity, retention factors and the identification of unknowns using chromatographic standards.
- Extraction[20,21]: Students isolated lemon peel extract and clove extract as sample solutions using 91% isopropanol as a solvent. The lemon peel extract contains limonene. Clove oil extract contains eugenol. Both extract solutions are tested with potassium permanganate solution to show the presence of an olefin in the extracted solution as the alkene reaction forms a syn-diol plus a MnO_2 precipitate. The lab experiment emphasizes polarity, extraction as a purification technique and visual identification of alkenes through the $KMnO_4$ reaction.

5.7.2 LO2: Common Organic Reaction Mechanisms

A second learning objective for this course was student familiarity with common reaction mechanisms. Two of the previously described laboratory techniques experiments also referenced reaction mechanisms.

- The Acids and Bases using Red Cabbage pH Indicator experiment discusses nucleophiles and electrophiles with identification of reaction components as Lewis acids or bases.
- The Extraction experiment reviews the addition of potassium permanganate to an alkene to form a syn-diol.

As well, four additional experiments supported student review of important organic reactions.

- Saturated and Unsaturated oils[22-24]: This experiment uses iodine tincture to assess saturation of unknown oil samples. The halogenation of alkenes reaction is discussed, and students are tasked to predict products of example compounds using correct stereochemistry.
- Making Polymers[25,26]: Students make the borax-glue based bouncy ball as an example of polymerization. The experiment discusses chain-growth and step-growth polymerization reactions, along with cross-linking events. Students are tasked to recognize common polymers with their uses in our society, and to predict the polymerized product when given sample reactants.

- Preparation of Soap[27,28]: Students create their own soap product using a provided oil mixture containing coconut, olive oil and lard. The saponification reaction is discussed along with characteristics that create an effective soap solution. Upon completion of the experiment, students test the pH of their soap using their red cabbage indicator solution saved from a previous experiment.
- Crossed Aldol Condensation[29,30]: Students perform a crossed aldol condensation reaction using cinnamaldehyde and acetone as reactants. The highly colored product precipitates quickly and students are able to filter and dry the crystalline product. Worksheet questions emphasize predicting the products for potential reactants to reinforce the reaction mechanism and support identification of acidic hydrogens.

5.7.3 LO3: Stereochemistry and Three-Dimensional Visualization of Organic Molecules

The remaining four assignments for the laboratory course were ChemSketch[31] focused worksheets developed by the authors to help students visualize organic structures in three dimensions. Two of these worksheets (Alkyl Halides & S_N1/S_N2 Reactions and Electrophilic Aromatic Substitution) also support the second learning objective of understanding reaction mechanisms. The primary focus of these activities was to allow students to visualize key concepts

in a particular reaction mechanism like the inversion of stereochemistry in the S_N2 reaction or the directing effects of substituents in the EAS reaction. By having a 3-D model in front of them, the students are provided additional insight into more difficult concepts like steric bulk or steric congestion that can account for many of the mechanistic pathways in organic chemistry.

- ChemSketch Worksheet- Molecular Modeling of Cyclohexane and Substituted Isomers: ChemSketch worksheet for visualization of the cyclohexane chair configuration and the spatial relationships of axial and equatorial substituents. Students are tasked to explain the relative stability of isomers related to equatorial or axial positioning.
- ChemSketch Worksheet- Stereochemistry: This exercise details the Cahn-Ingold-Prelog ranking system for chiral carbons and practices ranking compounds as R or S configuration. Visualization of example compounds supports students to understand chirality and the nomenclature system.
- ChemSketch Worksheet- Alkyl Halides and S_N1/S_N2 Reactions: The worksheet emphasizes the substitution reaction mechanisms for alkyl halides and tasks students to predict stereochemistry of products. Strength of nucleophile and alkyl halide size are emphasized to support understanding of these reaction mechanisms along with impact on product structure.

- ChemSketch Worksheet- Electrophilic Aromatic Substitution: The reaction mechanism for EAS and the directing effects of various substituents are explored. Resonance structures are emphasized in predicting potential products.

5.8 Student Performance

This research study was approved through our institutional IRB Board (IRB # 15-024) and all students included in results signed consent forms for the research study. Students were provided surveys after every 4 experiments (see Supporting Information, Instructor Resources: Student Surveys), and the data was collected and analyzed. Survey and Lab Report grade results have been tabulated into the figures and tables in this section, to reflect student attitudes and confidence with course learning objectives. The data presented represents two of the three semesters that these online labs were offered.

For Figure 5.3, we compared the final grades of all female students versus the final grades of all male students (p=0.5439, Student's t-test, two-tailed). For our particular cohort of students, the higher number of 0 grades lowered the final letter grades of our male students (median female= 90.0, male=84.8). It has been reported by Trueman[32] and Macan[33] that female students scored higher on time management scales, self-reported higher time-management skills, as well as reported higher utilization of daily planning to stay on top of assignments,

and the authors speculate this may have been a factor with overall grades in this course. Grades of “0” were included in these calculations and reflect a student not turning in assignments, as they contributed to the students’ final grades.

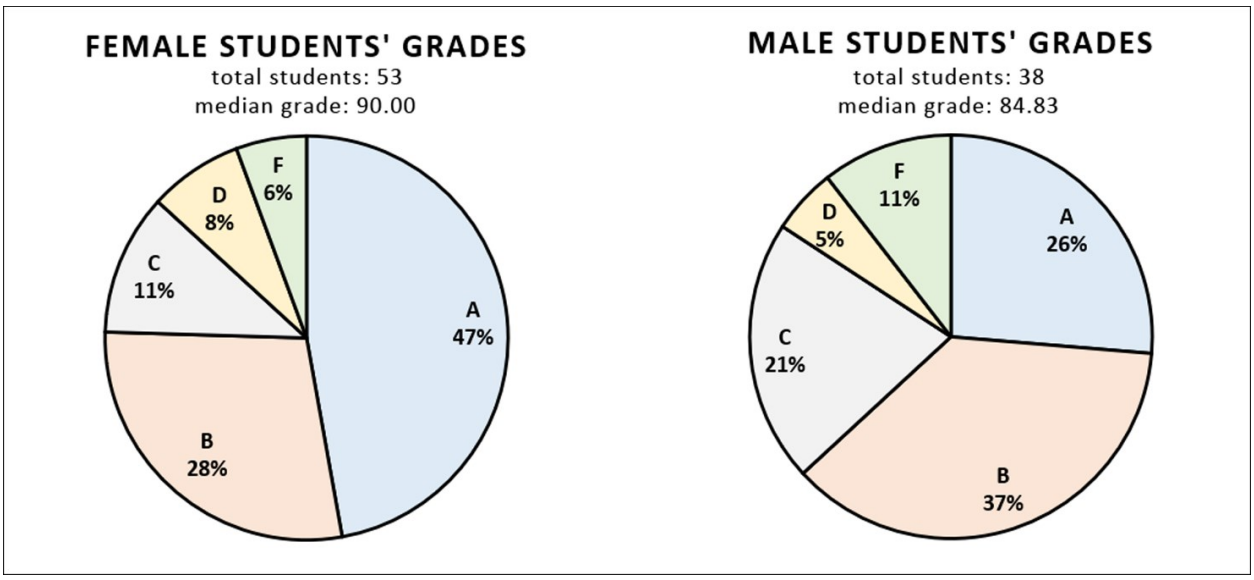


Figure 5.3 Final student grades, separated by gender

Table 5.2 provides the average student worksheet grades, separated by Learning Objective, gender, and semester. Grades of “0” were not counted in these calculations, as that signified that a student did not turn the assignments in, and we were interested in using this comparison to determine whether a student learned the objective upon attempting the assignment. The student surveys collected reflected frustration with the ChemSketch instructions and ease of use, and another goal when compiling this data was to determine whether grades were higher moving from Summer 2014 to Spring 2015 as we updated

and improved instructions between semesters to attempt additional support for students using the computer program.

Comparing grades from both semesters, learning objective 2 (LO2; support understanding of common organic reactions) and learning objective 3 (LO3; support student practice with stereochemistry and the three-dimensional visualization of organic molecules) demonstrated improvements after the previously mentioned changes were implemented between semesters. LO3 had a lower average grade among the students, as well as a higher rate of assignments not turned in, especially among male students with 21.2% of assignments turned in receiving 0 grade. The percent of 0's for all LO's was reported according to the total responses (including 0's) split by gender.

Table 5.2 Comparison of Average Student Worksheet Grades by Learning Objective and Gender

Learning Objectives	Total Assignments	**Performance Measures, Female Students**				**Performance Measures, Male Students**			
		Grade of 0		Average Grade[a]	SD[b]	Grade of 0		Average Grade[c]	SD[b]
		N[a]	%[a]			*N*[c]	%[c]		
Summer Semester, 2014									
LO1	104	0	0	92.88	3.23	5	9.6	89.04	4.63
LO2	104	1	1.9	84.26	3.97	3	5.8	83.21	4.15
LO3	52	3	11.5	67.28	9.81	6	23.1	71.63	7.62
Spring Semester, 2015									
LO1	264	6	3.8	91.33	3.80	12	11.5	91.06	3.40
LO2	264	15	9.4	88.69	3.71	12	11.5	88.29	3.47
LO3	264	27	16.9	79.79	6.40	22	21.2	77.10	7.35

[a]The total number of female students was 13 for the 2014 summer semester and 40 for the 2015 spring semester. [b]$p < 0.001$; chi-squared goodness of fit comparing LO3 to LO2, which had a higher number of assignments receiving a grade of 0. [c]The total number of male students was 13 for the 2014 summer semester and 26 for the 2015 spring semester.

For Table 5.3, survey results were tabulated to determine the students' overall feelings towards the lab experience. The majority of students reported that the instructions were generally clear, did not take an unreasonable amount of time, and that they would recommend the online lab experience to other students. Open-ended feedback was also given by students and has been compiled and taken into consideration by the instructors of the course.

Table 5.3 Survey results from Summer 2014 & Spring 2015

Questions for Student Response	Total Responses	Yes		No	
		N	%	*N*	%
Did you find the instructions clear?	229	194	85	35	15
Did you feel these labs related to your lecture material in CH 2503?	224	150	67	74	33
Did the experiments take too much time?	231	58	25	173	75
Would you recommend this lab experience to another student?	220	156	71	64	29
Did these labs help you better understand organic chemistry?	226	139	62	87	39

Figure 5.4 was compiled from the student surveys as well, in which the students were asked to pick their favorite and least favorite experiment from each set of four experiments (1-4, 5-8, 9-12). These results were supported by their feedback about difficulties using the ChemSketch software, which is further discussed in the Results and Discussion section.

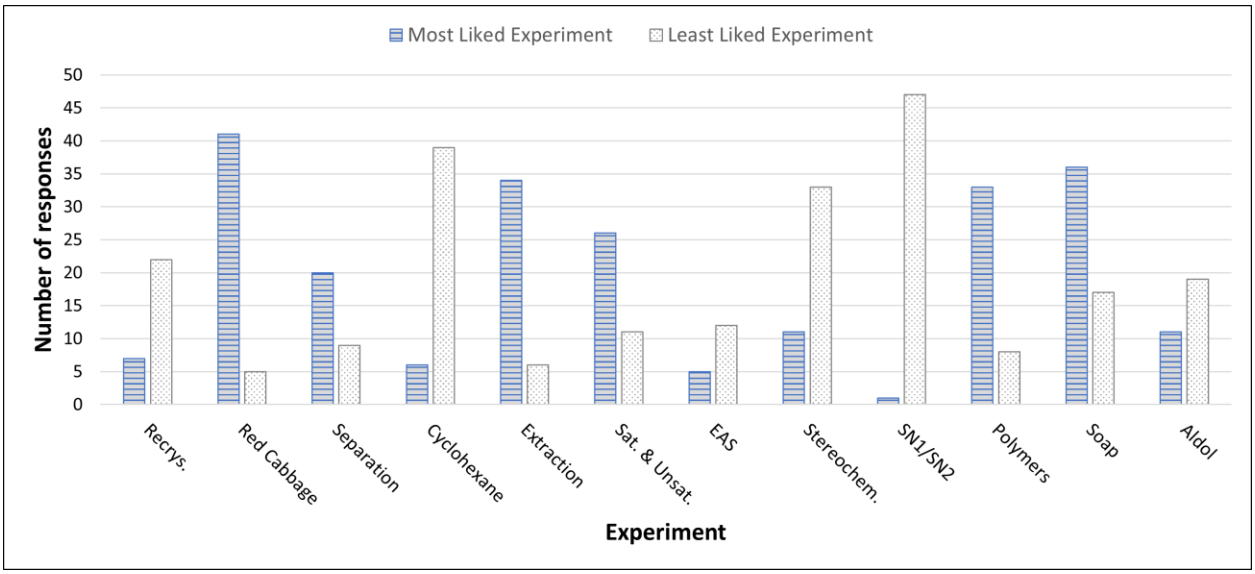


Figure 5.4 Survey results of students' favorite and least favorite experiments

5.9 Results and Discussion

In general, students were positive about the flexibility of the laboratory schedule and they enjoyed many of the experiments. Frustrations included the expectation that additional grocery items were needed for the experiments and a universal, all-encompassing hatred for ChemSketch. Using a Chi-Squared Goodness of Fit analysis, we found that the number of not submitted assignments (0 grade assignments) was far greater on LO3 than on LO1 or LO2 (p value< 0.001). While no statistical difference between male/female grade averages was noted, the prevalence of 0 grades on LO3 might have impacted the overall letter grades as female students had fewer 0 grades and a higher median grade for the course. The frustrations surrounding ChemSketch appeared to be that the program was too difficult for students to manage and even the authors' repeated modification of student instructions did not address their concerns. The authors note that a variety of newer visualization programs are now available online (such as Molsoft[34] and ChemDoodle[35]) which might better serve student needs and be easier for them to navigate.

Alongside of the already mentioned visualization programs, further relevant work has taken place in the field as a response to COVID-19's distance learning needs, leading to more methods of visualization that can help students understand hard-to-visualize concepts. Lau et al.[36] developed an app that is capable of molecular modeling that works alongside of activity worksheets which trigger augmented reality models on students' cell phones. Zhao et al.[37] created a molecular simulation tool called Manta, which uses virtual reality to allow

students to explore molecular structures and chemical reactions. Both programs were reported as receiving favorable responses from the students who used them and suggest that these applications could be used in conjunction with distance-learning labs in the future.

The frustration that students mentioned regarding additional grocery items reflected the authors' assumptions on kitchen supplies. We presumed that most students would have access to a basic kitchen with common tools. Dormitory kitchens, however, provide major appliances such as stove and refrigerator but typically provide no containers, pans or flatware. The authors also note that an additional avenue that instructors may wish to explore is the addition of a kitchen scale to applicable experiments. They can be purchased inexpensively and added to the lab kits, providing the students with additional post lab value that include weighing their created products, calculating percent yields, and understanding wet vs. dry weight. Future users of this kitchen-based course might want to consider adding in more basic supplies to the packed kit to support students using a limited dormitory kitchen.

The photo diary upload was a valued addition to this laboratory course. Instructors could follow student progress with the experiment uploads and quickly recognize student difficulties. As well, student comments reflected that students often took pride when their experiment worked as designed with photo evidence to showcase their results, which could lead to students feeling more engaged in the experiments when aiming for the best photo results.

5.10 Conclusion

This at-home laboratory course served our university's needs for three semesters, until an appropriate lab space became available on campus. With supervised lab space available, the course was adjusted back into an on-campus laboratory environment to minimize safety concerns. As institutions navigate the goals of student learning through online or independent courses, the need for self-paced hands-on lab courses will grow. This at-home laboratory experience can support those goals for introductory organic students.

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APPENDIX A

CHAPTER VI SUPPORTING INFORMATION

A.1 Supporting Information - Table of Contents

A.2 Instructor Resources

A.2.1 Introduction

The Instructor Resources section of this Supporting Information includes all the information needed to prepare for these lab experiments. There is a chemical purchasing list, lab kit packing list, checklist of all materials needed for each experiment, a recipe list to prepare the needed solutions, a student shopping list, and full walkthroughs for the wet lab experiments.

Careful thought should be put into the student shopping list, as instructors should pay attention to what materials students may or may not have. Dorm kitchens have very little when compared to a home kitchen, and it may be useful to include more materials that we had considered “standard”.

A.2.2 Chemicals Purchasing List

Cinnamaldehyde, CAS: 104-55-2

Acetone, CAS: 67-64-1

NaOH, CAS: 1310-73-2

$KMnO_4$, CAS: 7722-64-7

A.2.3 Packing List

Experiment 1: Recrystallization of Aspirin

Materials	Chemicals
Two 50-mL test tubes	Aspirin tablets (12 tablets in a Ziploc bag)
Plastic funnel	

Experiment 2: Acids & Bases using Red Cabbage pH Indicator Solution

No materials or chemicals needed

Experiment 3: Separation of Food Dyes by Chromatography

Materials
Plastic pipet

Experiment 4: Molecular Modeling of Cyclohexane & Substituted Isomers

No materials or chemicals needed

Experiment 5: Extraction

Materials	Chemicals
Two plastic pipets	$KMnO_4$ solution (~10 mL in a dropper bottle)
One 50-mL test tube	
Two tablespoons of ground cloves (in Ziploc bag)	
Plastic funnel	
Two 15-mL test tubes	

Experiment 6: Saturated & Unsaturated Oils

Materials	Chemicals
Three 15-mL test tubes	Oil A (~10 mL in vial) Oil B (~10 mL in vial) Oil C (~10 mL in vial) Iodine tincture solution (~10 mL in dropper bottle)

Experiment 7: Electrophilic Aromatic Substitution

No materials or chemicals needed

Experiment 8: Stereochemistry

No materials or chemicals needed

Experiment 9: Alkyl Halides and S_N1/S_N2 reactions

No materials or chemicals needed

Experiment 10: Making Polymers

Materials	Chemicals
Plastic spatula	Elmer's Glue (27-28 mL in a 50-mL test tube) Borax (4.8 grams in Ziploc bag)

Experiment 11: Preparation of Soap

Materials	Chemicals
250-mL plastic beaker	Oil mixture (in a plastic jar)
Plastic spatula	Lye solution (in a 50-mL test tube)
One 15-mL test tube	
Plastic pipet	

Experiment 12: Crossed Aldol Condensation

Materials	Chemicals
One 50-mL test tube	Cinnamaldehyde (~10 mL in vial)
Plastic spatula	Acetone (~10 mL in vial)
Funnel	50% NaOH solution (~10 mL in dropper bottle)
Two plastic syringes	

A.2.4 Lab Kit Checklist

Materials

- Four 50-mL test tubes with caps
- Six 15-mL test tubes with caps
- Plastic funnel
- Two plastic 3-mL syringes
- Three plastic spatulas (look like straws)
- Four plastic pipets
- One 250-mL beaker
- Safety goggles
- Two pairs of nitrile gloves

Chemicals

- Oil A in a glass vial
- Oil B in a glass vial
- Oil C in a glass vial
- Cinnamaldehyde in a glass vial
- Acetone in a glass vial
- Iodine tincture solution in a dropper bottle
- 50% NaOH solution in a dropper bottle
- $KMnO_4$ solution in a dropper bottle
- Aspirin tablets in a Ziploc bag
- Ground cloves in a Ziploc bag
- Borax in a Ziploc bag
- Oil mixture in a plastic jar
- Lye solution in a 50-mL test tube
- Elmer's glue in a 50-mL test tube

A.2.5 Student Shopping List

These items are needed for the CH 2501 lab experiments and are not included in the lab kit:

A pack of coffee filters

4 clear glass jars with lids (½ pint, pint, or quart size will be fine); mason jars or Tostitos® jars work great

A sink with hot and cold water

A stove

A refrigerator

Ice

Dishwashing or hand soap

Paper towels

Butter knife, sharp knife, teaspoon, and tablespoon

Bowl

Small cooking pot (to boil water)

One 12-16 oz. bottle of 91% isopropyl alcohol (rubbing alcohol); it has to be 91%

A paper plate

One fresh lemon

Some rubber bands

One head of red cabbage

Some household chemicals (see Acid/Base experiment for more details)

Measuring cup

Cornstarch

Small Ziploc bags

Paper clip or toothpick

Ruler

Small bag of M&Ms or Skittles

Table salt

Food coloring (4 colors)

A.2.6 Recipe List

Experiment 1: Recrystallization of Aspirin

Impure Solid: Aspirin (Equate)

Experiment 5: Extraction

0.1 M $KMnO_4$ solution: 1.58 g of $KMnO_4$ diluted to 100 mL with water
OR 15.8 g of $KMnO_4$ diluted to 1 L with water

Experiment 6: Saturated & Unsaturated Oils

Iodine tincture solution: 1 mL of iodine tincture (CVS: 2% iodine & 2.4% NaI) in 9 mL of water

Oil A: Baby oil (Johnson's)

Oil B: Canola oil (Clover Valley)

Oil C: Vegetable oil (Great Value)

Experiment 11: Preparation of Soap

Oil mixture: 30 grams coconut oil (LouAna), 30 grams lard (Armour), & 40 grams olive oil (Best Choice)
(Melt them together on low heat)

Lye solution: 14.3 grams NaOH in 35 mL water

Experiment 12: Crossed Aldol Condensation

50% (w/w) NaOH solution: 50 g of NaOH in 50 mL of water
OR 100 g of NaOH in 100 mL of water

A.2.7 Student Surveys

Survey #1 **Name:**

This survey is designed for you to give feedback on the first set of four experiments:

Recrystallization of Aspirin
Acids and Bases using Red Cabbage pH Indicator
Separation of Food Dyes by Chromatography
Molecular Modeling of Cyclohexane

You will receive credit for this assignment if you simply fill it out. Please be honest! Thank you.

Survey Questions for the first four experiments:
1. Did you find the instructions clear? Were any instructions confusing for you?

2. Did you feel these labs related to your lecture material in CH 2503? Please explain your answer.

3. Which lab experiment did you like most? Explain why.

4. Which did you like least? Explain why.

5. Did the experiments take too much time? Explain your answer.

6. Would you recommend this lab experience to another student?

7. Did these labs help you better understand organic chemistry? Explain your answer.

8. Please give us any other comments or feedback that will help us improve.

Survey #2 **Name:**

This survey is designed for you to give feedback on the second set of four experiments:

Extraction
Saturated and Unsaturated Oils
Electrophilic Aromatic Substitution
Stereochemistry

You will receive credit for this assignment if you simply fill it out. Please be honest! Thank you.

Survey Questions for the first four experiments:
1. Did you find the instructions clear? Were any instructions confusing for you?

2. Did you feel these labs related to your lecture material in CH 2503? Please explain your answer.

3. Which lab experiment did you like most? Explain why.

4. Which did you like least? Explain why.

5. Did the experiments take too much time? Explain your answer.

6. Would you recommend this lab experience to another student?

7. Did these labs help you better understand organic chemistry? Explain your answer.

8. Please give us any other comments or feedback that will help us improve.

Survey #3 **Name:**

This survey is designed for you to give feedback on the third set of four experiments:

Alkyl Halides & S_N1/S_N2 reactions
Making Polymers
Preparation of Soap
Crossed Aldol Condensation

You will receive credit for this assignment if you simply fill it out. Please be honest! Thank you.

Survey Questions for the first four experiments:
1. Did you find the instructions clear? Were any instructions confusing for you?

2. Did you feel these labs related to your lecture material in CH 2503? Please explain your answer.

3. Which lab experiment did you like most? Explain why.

4. Which did you like least? Explain why.

5. Did the experiments take too much time? Explain your answer.

6. Would you recommend this lab experience to another student?

7. Did these labs help you better understand organic chemistry? Explain your answer.

8. Please give us any other comments or feedback that will help us improve.

A.2.8 Wet Lab Walkthroughs

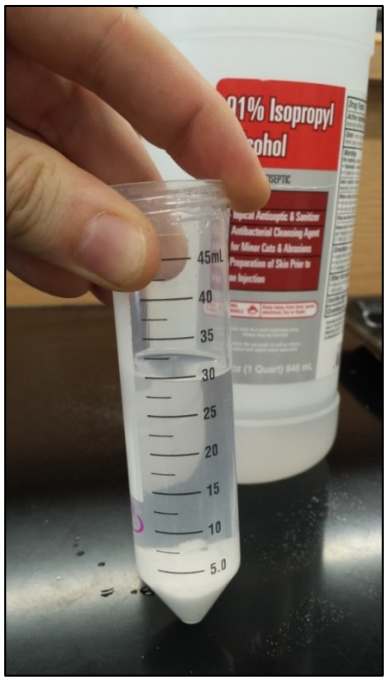
Experiment 1: Recrystallization of Aspirin
Place six aspirin tablets on a paper plate then crush them with a metal tablespoon (or teaspoon).

Six aspirin tablets before crushing

Crushed aspirin tablets

Carefully transfer the aspirin powder to a 50-mL test tube. Fill the test tube up to the 30-mL mark with 91% isopropanol.

Aspirin powder in test tube



Adding isopropanol to test tube

Heat up a cooking pot half-full of water on the stove (turn the dial to MED). DO NOT BOIL THE WATER. While the water is heating up, fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of a clean 50-mL test tube. Place this filtration setup (coffee filters, funnel, & test tube) in a jar to keep it upright.

Heating up water in a pot

First filtration setup

Once the pot of water is hot, submerge the bottom half of the aspirin-containing test tube into hot water. You want to heat the aspirin-containing test tube in the hot water. DO NOT CAP THE TEST TUBE. Swirl the test tube for 10-15 minutes in the hot water bath. Some of the solid should dissolve and the solution may appear cloudy.

Heating aspirin solution

After solution was heated

While the solution is still hot, pour it into your filtration setup that you made earlier. You shouldn't see any solid particles in the filtrate. **Take a picture of the filtrate.**

Hot filtering the solution

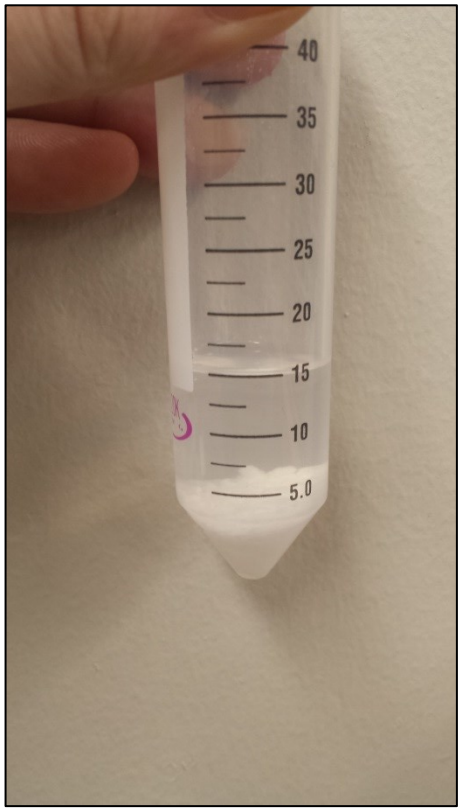
Clear filtrate

Once the filtration is done, add one tablespoon of water to the resulting liquid (filtrate). Let the mixture stand for 10 minutes then put the cap on the test tube. Place the test tube in a jar of ice then put the jar in the refrigerator for 12-24 hours.

Adding one tablespoon of water

Solution in a jar of ice

After the solution has cooled for 12-24 hours, solid crystals should have appeared at the bottom of the test tube. **Take a picture of the crystals that grew inside the test tube.** Construct another filtration setup similar to the one you made before. Fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of a jar.

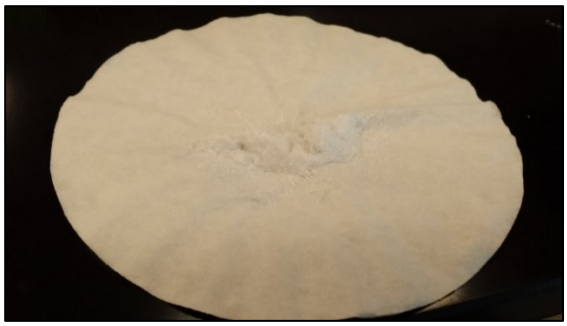


Crystals of aspirin after cooling

Second filtration setup

Filter the aspirin solution using your filtration setup. You can use cold water to remove any solid that sticks to the test tube. When it's done filtering, lift the filter paper out and lay it on a few paper towels. Allow the solid to air dry for a few hours. **Take a picture of your dry solid.**

Product still damp



Dry product

Experiment 2: Acids & Bases using Red Cabbage pH Indicator Solution Walkthrough

Making the pH indicator:
Chop about 2 cups of red cabbage into strips (about ¼ of a head) and put in medium sized bowl.

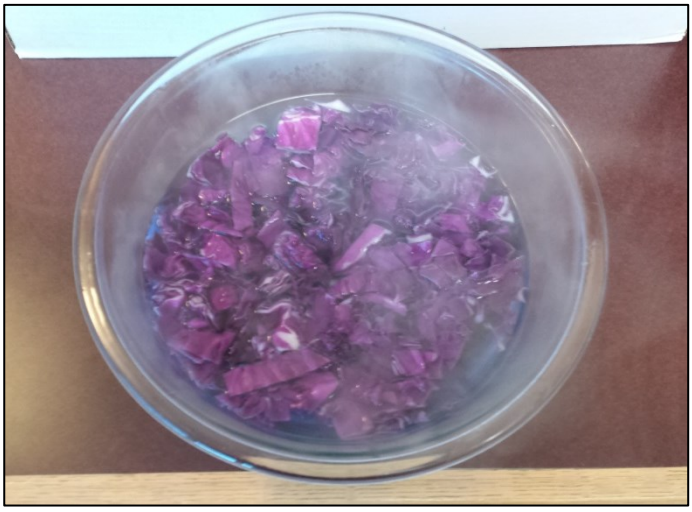
One quarter of a head of red cabbage

Two cups of chopped red cabbage

Two cups of chopped red cabbage in a bowl

Boil 4 cups of water on a stove. Pour the boiling water over the chopped cabbage and let it steep for 10-15 minutes. You want to get a dark, rich color in the water. **Take a picture of your soaking cabbage.**

Pouring hot water over cabbage



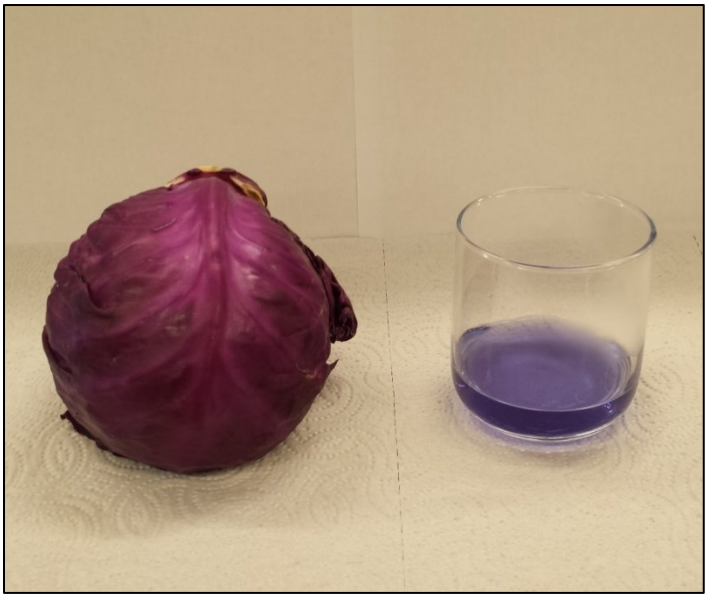
Steeping cabbage

When it is cool, you can scoop the cabbage out so you just have colored extract in the bowl or filter the solution through a few paper towels. Gather up all of your solutions that you want to test. **Take a picture of all the items you gathered for the pH test.**

Filtering the cabbage solution

Items for testing

pH Testing:
Pour some of the extract into your four clear glasses. You just need enough to see the color well. Estimate the pH of your extract before adding anything (use the picture in the color table provided in the worksheet to estimate the pH). Pour an equal amount of each of your collected solutions into your clear glasses with the pH extract (one at a time). Estimate the pH of each solution by comparing color to the chart. **Take a picture of all 4 solutions for your photo logbook.** Pour your solutions down the sink (with plenty of water) and rinse the glasses out completely to repeat test with new solutions. Repeat the same procedure for the next 4 solutions. **Take a picture of all 4 solutions for your photo logbook.**



Cabbage indicator (nothing added)

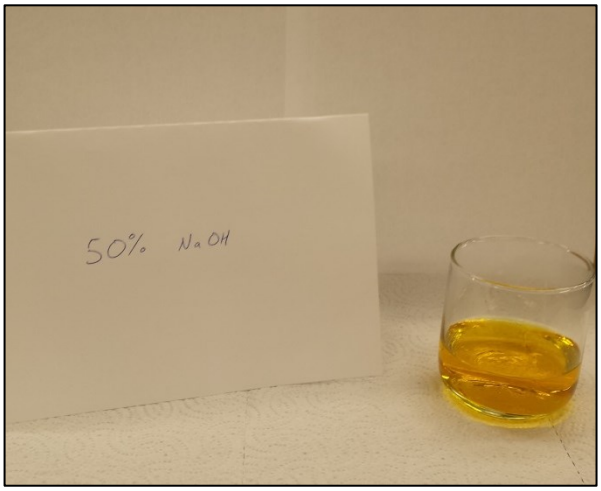
Solutions with Representative Colors and pH Values	Identity (from left to right)
	1. Cabbage extract by itself ; pH 6-7 2. Water; pH 6-7 3. Windex; pH 6-7 4. Fantastik; pH 9
	1. Pickle juice; pH 1 2. Lemon juice; pH 2 3. Sprite; pH 2 4. Orange juice; pH 2-3
	1. Mouthwash; pH 6 2. Shampoo; pH 5 3. Bodywash; pH 4 4. Dishwashing soap; pH 5
	1. Vinegar; pH 2 2. Baking soda; pH 9 3. Baking powder; pH 7-8 4. Seltzer water; pH 4
	1. White grape juice; pH 4 2. Liquid plumber; pH 11-12 3. Pine sol (ammonia); pH 9 4. Laundry bleach; pH 11-12

Nonfat powder milk

Aspirin tablet

Acetaminophen tablet

Stay Awake (caffeine) tablet



50% NaOH solution from Aldol Lab

Pepperoncini juice

Olive oil

Coffee

409 cleaner

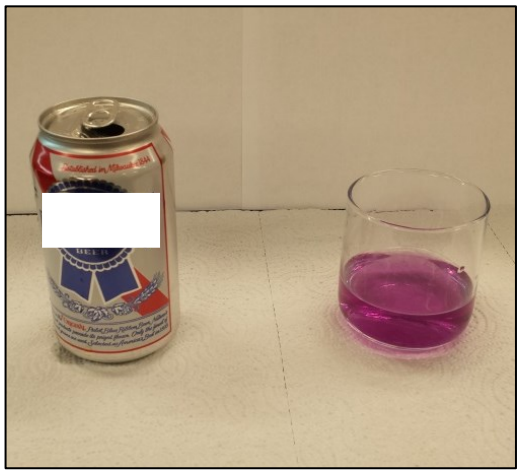
Powerade sports drink

Tide laundry detergent

Mean Green cleaner

Tums antacids

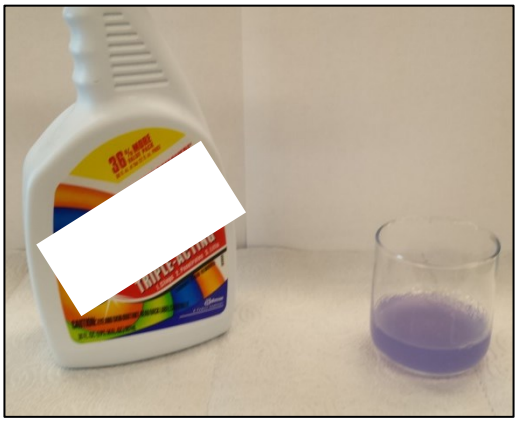
Apple juice



Beer

CLR (calcium, lime & rust) remover

Liquid sweetener



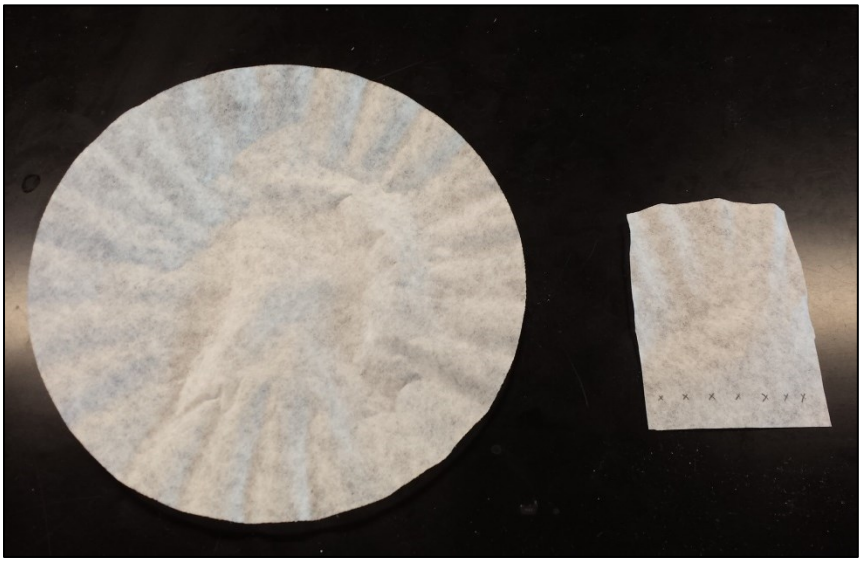
Shout stain remover

SAVE ½ CUP OF YOUR CABBAGE INDICATOR SOLUTION: Put it in a sealed jar or plastic container and store it in the refrigerator. You will need it for the Soap experiment.

Cabbage indicator for Soap Experiment

Experiment 3: Separation of Food Dyes by Chromatography

Preparing the Coffee Filters:
Gently flatten out a coffee filter on the countertop. Cut the filter in half then cut the rounded edges off the sides. Draw seven light "X" marks on the bottom cut edge with a PENCIL. You should place your marks about 1.0 cm from the bottom edge of the filter paper.

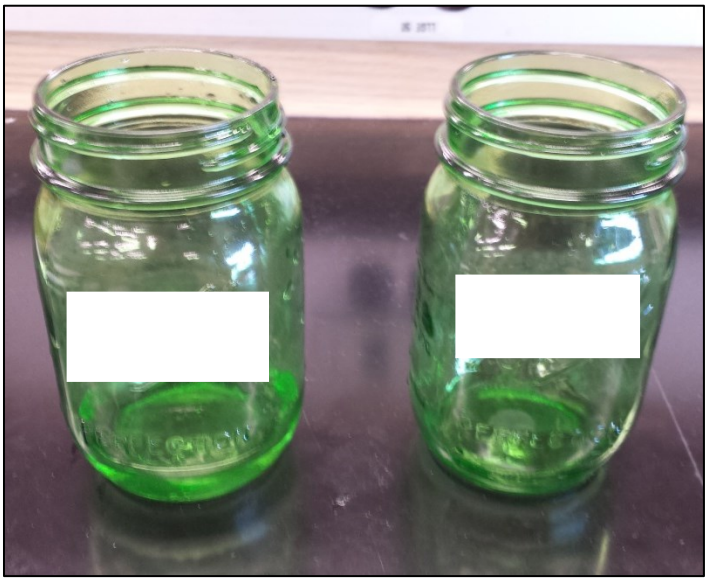


Cutting and marking the coffee filter

Preparing the M&Ms or Skittles for Chromatography:
Pull out 3 colors of M&Ms or Skittles and place each color in its own plastic cup. Add 6 drops of 91% isopropanol and 6 drops of water to each cup with a plastic pipet. Soak each candy for about 15 minutes. Swirl the mixture occasionally to get the maximum amount of dye off the candy. You want to get a rich concentrated color in your isopropanol solution. **Take a picture of the M&Ms or Skittles soaking the isopropanol solution.**

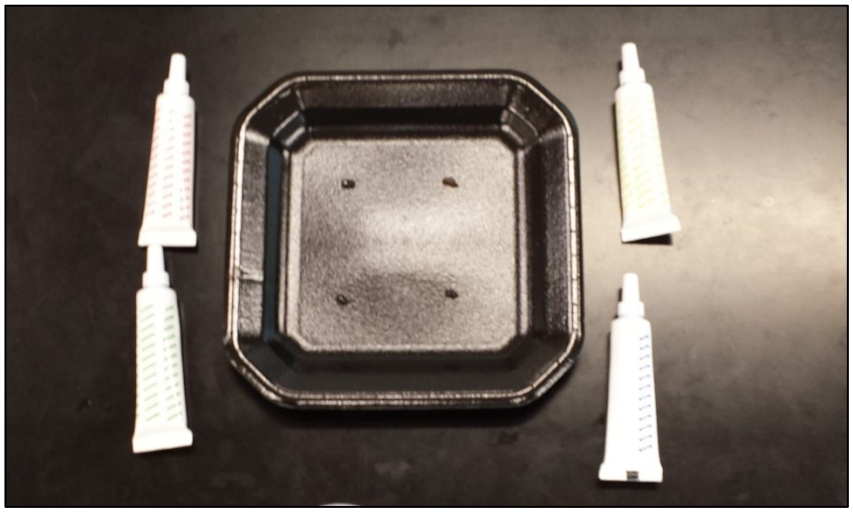
Soaking M&Ms in isopropanol solution

Preparing Chromatography Chamber:
Mix 1.5 tsp of table salt (NaCl) in a ½ cup of water in a clean jar. Make sure all the salt dissolves before use. Pour some of the salt solution onto a second jar; you barely want to coat the bottom of the jar with the salt solution. This second jar will be your chromatography "chamber".



NaCl solution (Left) & chromatography chamber (right)

Preparing Food Dyes for Chromatography:
Squeeze out a small drop of the 4 food dyes onto your paper plate. Check your box of food dyes to identify the dye colors included in your box. The most common ones are Red #40, Yellow #5 and Blue #1. Write down your dye colors on your worksheet.

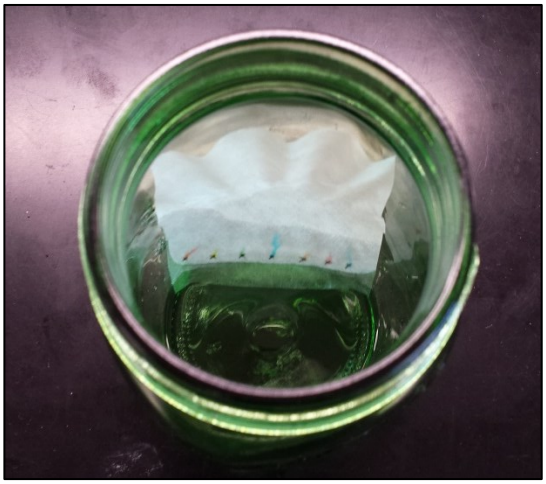


Four different food coloring dyes on a paper plate

Running the Dye Samples:
Dip a clean toothpick or unfolded paper clip into each dye solution (the 4 four food dyes and 3 candy solutions) and dot each gently onto one of the "X" marks of your cut coffee filter. Place one color on to each "X" spot. You want to dot your sample multiple times (2-3 times) while each time having the spot remain as small as possible. Let the spot dry in between each application so that the spot can stay as small as possible. Wipe your toothpick clean (or get a new one) after each dye so you don't contaminate one spot with another.

Dyes on coffee filter

After all the colors are on the coffee filter, place the coffee filter in your chromatography chamber. Be careful that the solvent stays BELOW your “X” marks where your spots are marked and also make sure the cut edge of your coffee filter is down in the liquid. Allow your coffee filter to sit UNDISTURBED while the salt water moves up the sheet.



Solvent moving up the coffee filter

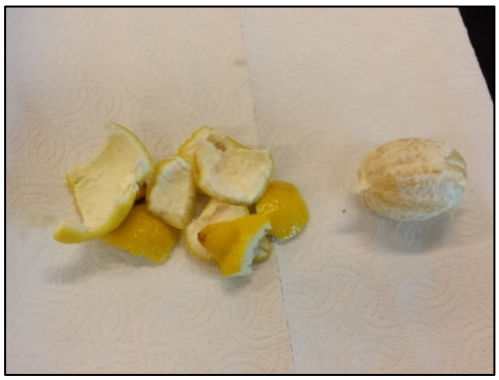
Watch and remove the coffee filter from solution when the solvent line ~ 1 cm from the top edge of the filter. Mark the edge of the solvent lightly with a pencil all the way across the filter. The solvent is very volatile and will begin to evaporate soon, so do not delay. Wait for your coffee filter to dry and then

gently flatten it out on the counter. Lightly circle all of the dye spots present on your plates with a pencil. Measure the R_f values for each of the different dye spots and record your data on your worksheet. When measuring R_f values, measure from the "X" mark to the leading edge of the dye spot then divide by the distance from the "X" mark to solvent front. Identify your unknown dyes. Match your unknown dye spots to the 4 known dyes from the food coloring. If they have the same R_f value, then they are a match. If you have a spot with an R_f value that does not match, list it as an unknown. **Take a picture of the coffee filter after running the dyes in the chromatography chamber.**

Dyes post-run
Left to right: Red 40, Yellow 5, Green (Mix of Yellow 5 & Blue 1), Blue 1, Orange M&M, Red M&M, & Blue M&M

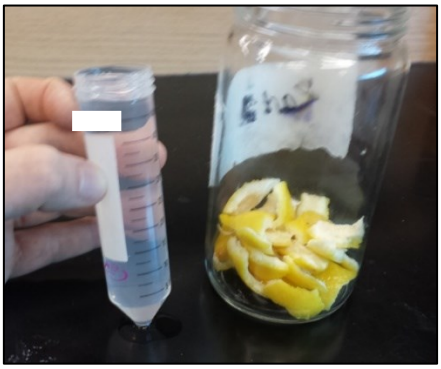
Experiment 5: Extraction

Creating the Lemon Peel Extract: Peel one lemon and discard the fruit's core. Tear the lemon peel into small pieces and place them into an empty jar.



Peeled Lemon

Lemon peel in jar



Measuring out isopropanol

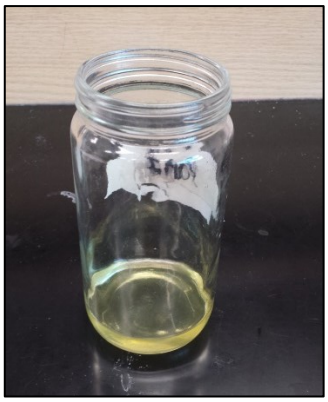
Using a 50-mL test tube, measure out 50 mL of 91% isopropanol and add it to the jar of lemon peels. Put the lid on the jar and shake it vigorously for 15-20 minutes. After shaking, let the lemon peel solution settle for 5 minutes. While you're waiting, fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of another empty jar.

Lemon peels in isopropanol

Filtration setup

Filtering the lemon extract

Filter the lemon peel solution using the filtration setup that you just constructed. The resulting liquid (filtrate) shouldn't have any solid pieces floating in it. Discard the used lemon peels. Set the lemon filtrate aside. **Take a picture of the lemon filtrate.**

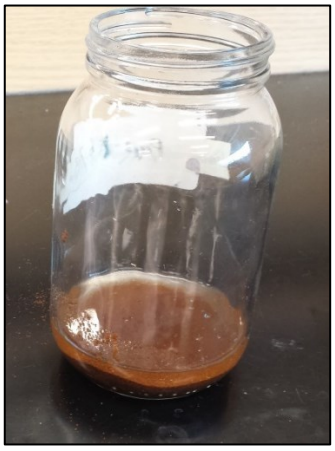


Lemon filtrate

Creating the Cloves Extract: Add two tablespoons of ground cloves to an empty jar. Using a 50-mL test tube, measure out 50 mL of 91% isopropanol and add it to the jar of ground cloves.

Adding cloves to the jar

Measuring out isopropanol



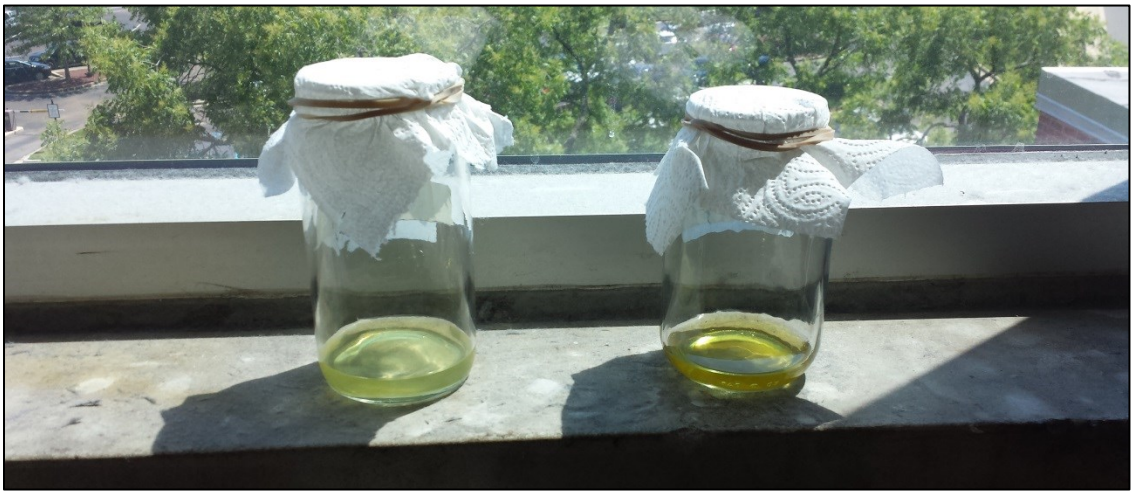
Ground cloves in isopropanol

Put the lid on the jar and shake it vigorously for 15-20 minutes. After shaking, let the clove solution settle for 5 minutes. Create a filtration setup like the one you made for the lemon peels. Fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of another empty jar. Filter the clove solution using the filtration setup. The resulting filtrate shouldn't have any solid pieces floating in it. Discard the used clove powder. **Take a picture of the clove filtrate.**

Filtering the clove extract

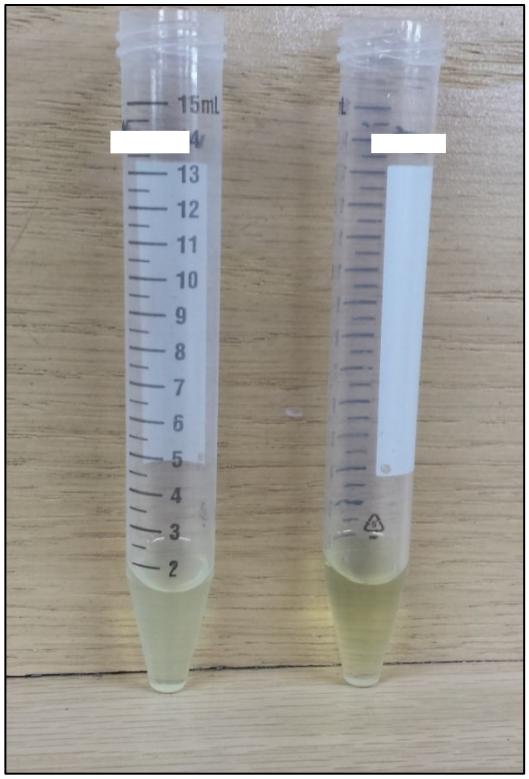
Clove filtrate

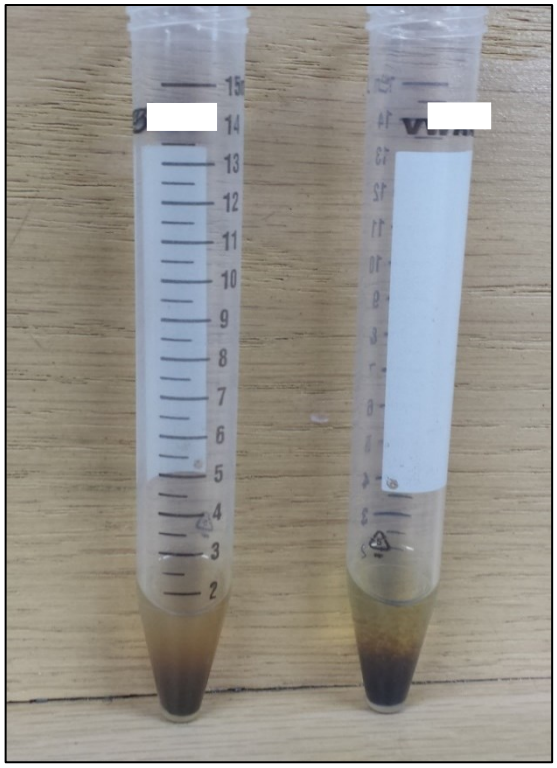
Evaporation & Testing: Place a paper towel over both jars containing your filtrates (lemon peel & cloves). You can secure the paper towel with a rubber band. The paper towel should fully cover the mouth of the jars to keep out solid debris. Let both jars sit in a window sill for 24 hours. Over the 24 hour period, some of the isopropanol will evaporate and your extracts will become more concentrated.



Both filtrates sitting in a window sill

After the 24 hour period, obtain two 15-mL test tubes. Using two different plastic pipets, place 2 mL of lemon peel extract in one test tube and 2 mL of clove extract in the other test tube. Add one drop of $KMnO_4$ solution to each test tube and swirl. Record what you observe happening in the test tubes after the $KMnO_4$ solution was added. Let the mixture sit for 5-10 minutes. **Take a picture of the test tubes after you let them sit for 5-10 minutes.**

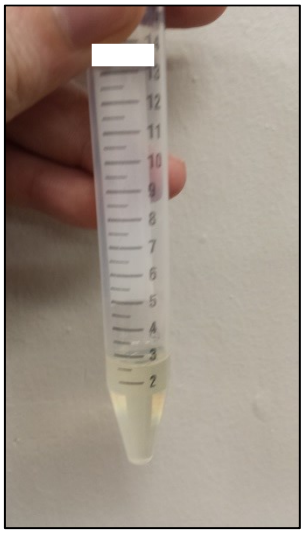




Lemon peel (left) and cloves (right) extracts After one drop of $KMnO_4$ solution was added

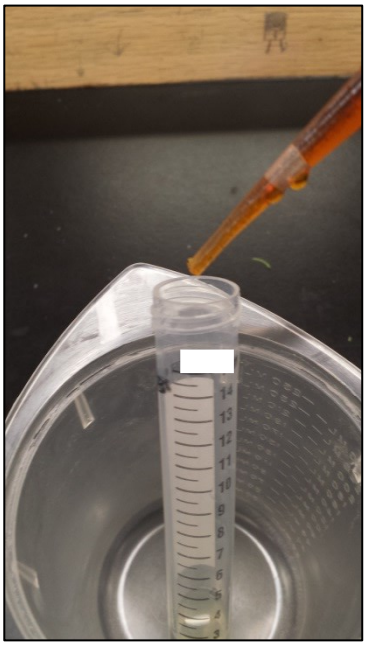
Experiment 6: Saturated & Unsaturated Oils

Label three 15-mL test tubes with the letters: A, B, & C. To test tube A, pour in ~2.5 mL of Oil A. To test tube B, pour in ~2.5 mL of Oil B. To test tube C, pour in ~2.5 mL of Oil C.

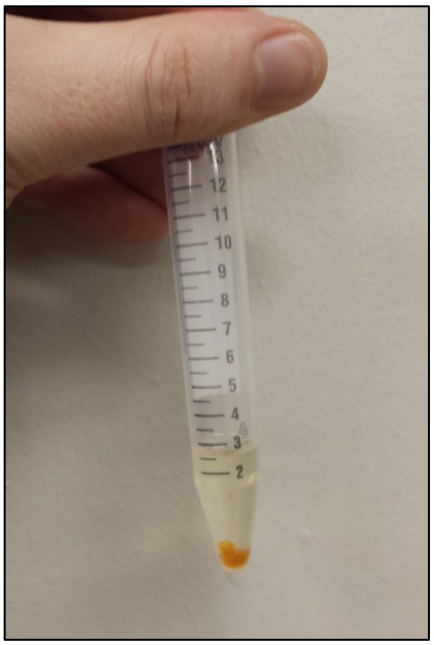


Oil in the test tube

To each test tube, add three drops of the iodine tincture solution. **DO NOT SHAKE THE TEST TUBES.** The iodine solution should sink to the bottom of the tubes.

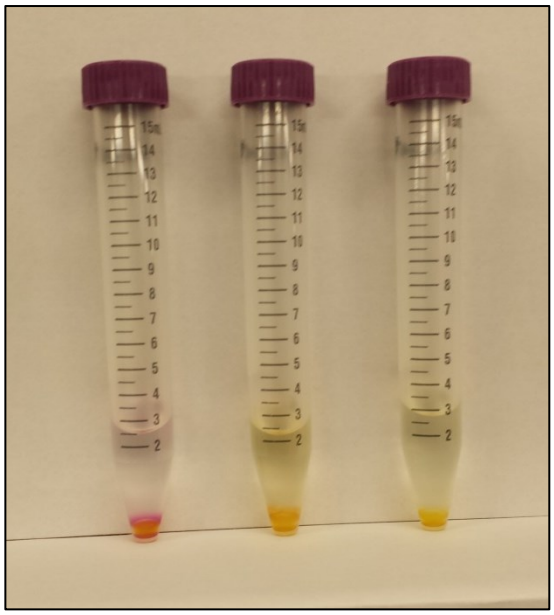


Adding the iodine solution

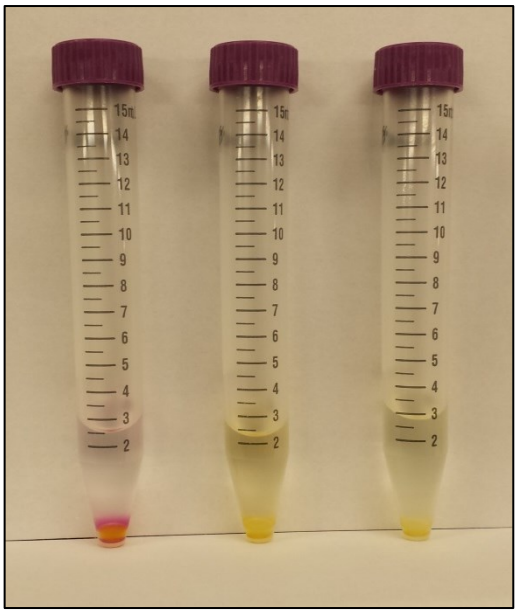


Iodine solution in oil

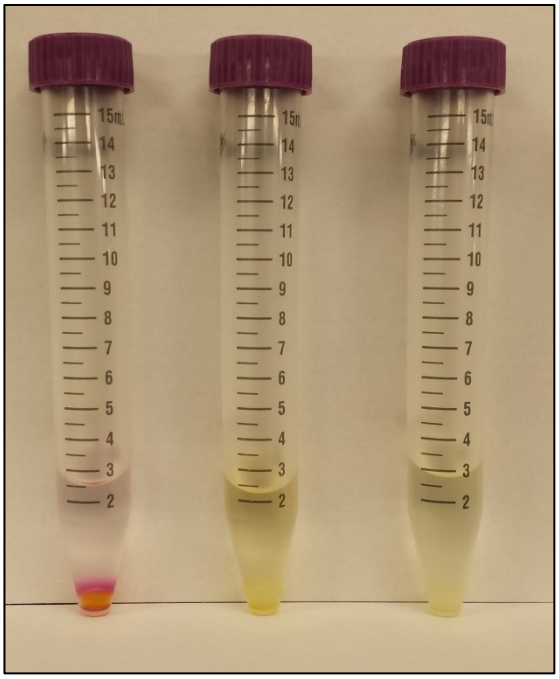
Observe all three test tubes over a one hour period. **Take pictures of all three test tubes after 10 minutes, 20 minutes, 30 minutes, and 1 hour.** After 1 hour, you can dispose of the oil solutions.



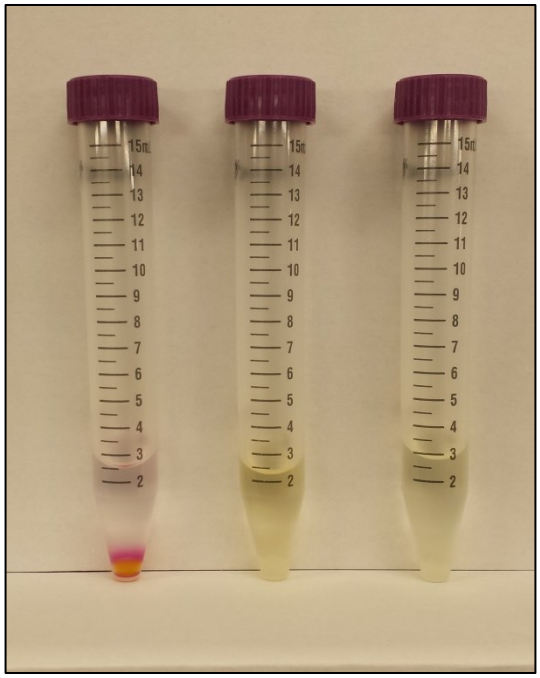
After 10 minutes (left to right: A, B, & C)



After 20 minutes



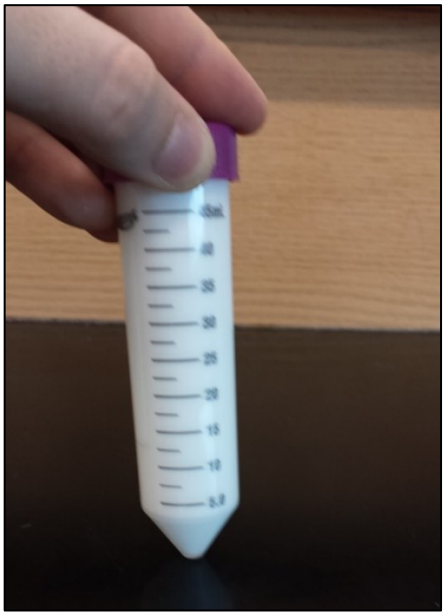
After 30 minutes



After 1 hour

Experiment 10: Making Polymers

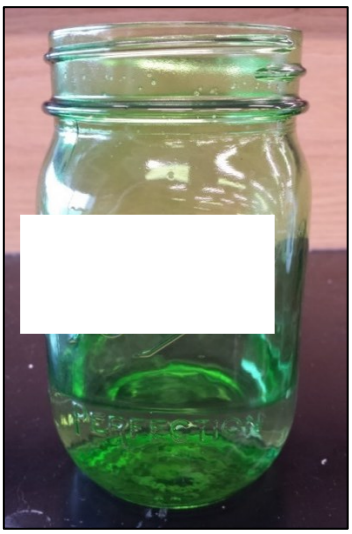
Making Glue Solution:
Open your test tube that contains the Elmer's glue. Add warm water to the 50 mL mark on the side of the tube. Cap the test tube and shake it well.



Glue solution (no water) **After adding water and shaking**

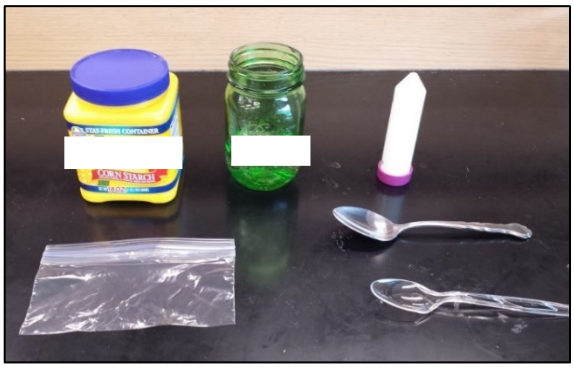
Making Borax Solution:
Pour all of your borax (in a Ziploc bag) into a small jar. Add ½ cup of warm water and stir it up with a plastic spatula. Make sure all the borax powder dissolves before use.

Borax powder in jar

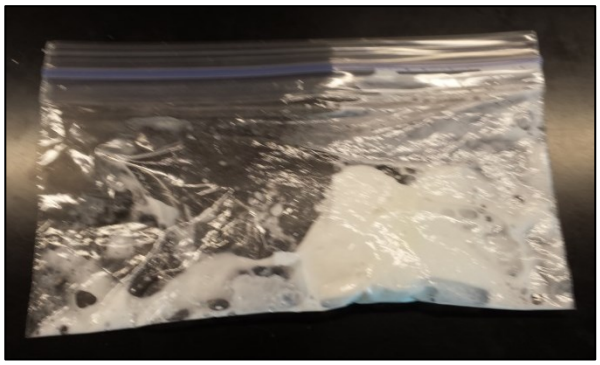


Dissolved Borax solution

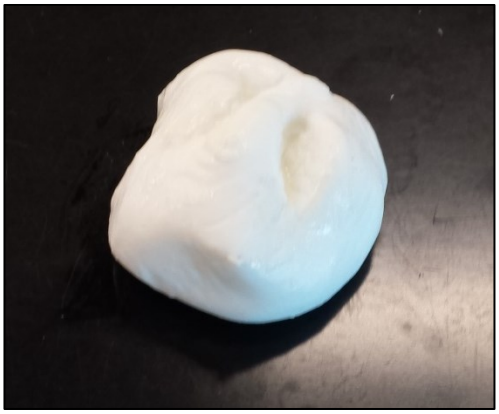
Making Polymers:
In a small Ziploc bag, place 2 tbsp of your glue solution and ½ tsp of your borax solution. Add food coloring if you want to make it look good (Caution: Food coloring may stain your hands or clothes). Mix or smush the polymer from your baggie and knead it for SEVERAL minutes. **Take a picture for your photo logbook once it forms a ball.** Slowly pull your polymer and record your observations. Abruptly pull polymer and record your observations. Roll the polymer into a ball and drop it. Record your observations. Repeat your experiment again; but this time, add 1 tbsp of cornstarch to the mixture. **Take a photo of the polymer ball** and record all your observations.



Gathered items



Mixing in Ziploc bag



Polymer ball (no cornstarch)

Polymer ball (with cornstarch)

Both polymer balls after sitting for ~15 minutes

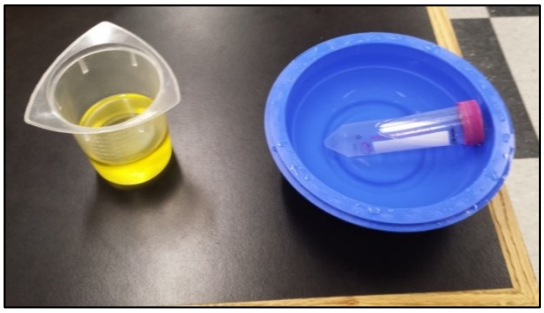
Experiment 11: Preparation of Soap

Pour the oil mixture into a small cooking pot. Lightly heat the mixture on the stove (turn the dial to LOW-MED). You'll notice that the cloudy oil becomes transparent as it heats.

Cloudy oil mixture before heating

Transparent oil mixture after heating

While the oil is heating up, place the test tube contain the lye solution in a bowl of hot water. DO NOT TAKE THE CAP OFF. You want use the hot water bath to heat up the lye solution inside the test tube. Once the oil mixture has become transparent pour it into the 250-mL beaker. Let the oil mixture cool down. You want the lye solution and the oil mixture to be about the same temperature (warm to the touch).



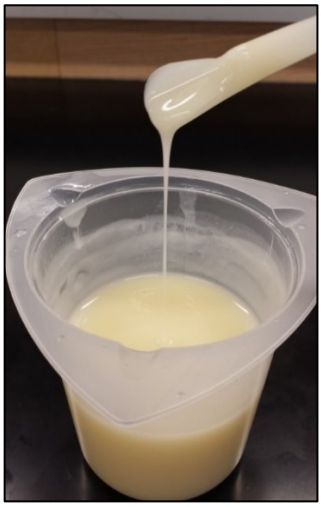
Warm oil (left) and warm lye solution (right)

Right after pouring lye solution in the oil

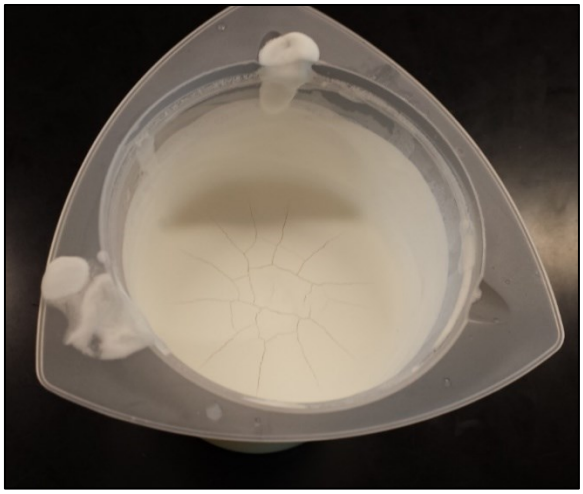
Once both solutions are about the same temperature, uncap the lye solution and pour it into the oil mixture in the beaker. Immediately after pouring the lye solution in, you'll notice that there are two separate layers of liquid. Use the plastic spatula to stir the mixture. You want the two layers to mix. As you mix, the solution will become cloudy. Stir the mixture periodically (every few minutes) for two hours. DO NOT LET THE MIXTURE SEPARATE INTO TWO LIQUID LAYERS. Over the two hour period, the solution will begin to thicken and the yellow color will start to lighten. It will have about the consistency of lotion. **Take a picture of your oil/lye mixture after you have stirred it for two hours.** After the two hours of mixing, let the mixture sit, undisturbed for 48 hours. The solution should harden over the 2-day period. **Take a picture of your hardened soap.**

Oil/lye mixture shortly after mixing

Oil/lye mixture one hour after mixing

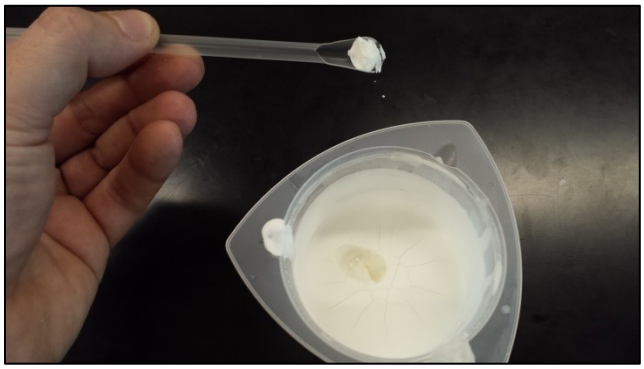


Oil/lye mixture after 2 hours of mixing

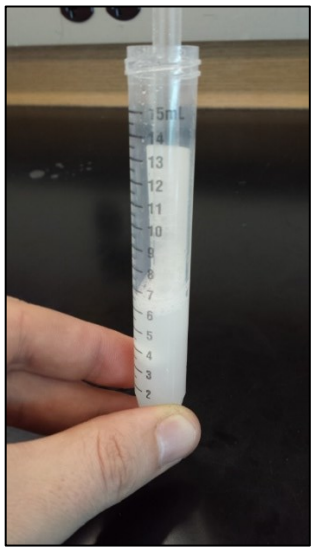


Hardened soap after two-day period

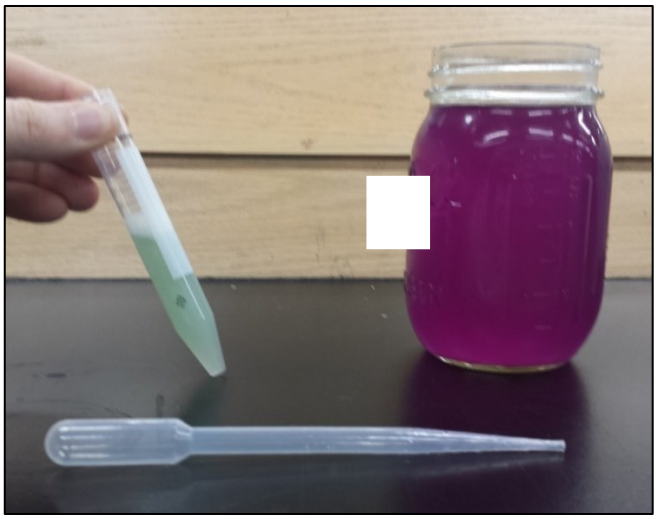
With a clean plastic spatula, take about a pea-sized amount of your soap and put it into a 15-mL test tube. Fill the test tube about half full of water and mix the soap thoroughly in the test tube. Using a plastic pipet, add 25-30 drops of your red cabbage indicator solution (from the pH experiment). Mix the solution well and determine the pH based on the color of the solution. **Take a picture of the colored solution after you added the red cabbage indicator.** If the pH is less than 10, it is safe to use. If the pH is greater than 10, the soap could be irritating to skin and should be disposed of. If the soap is safe to use, you can cut it into bars or disks using a butter knife.



A pea-sized amount of soap



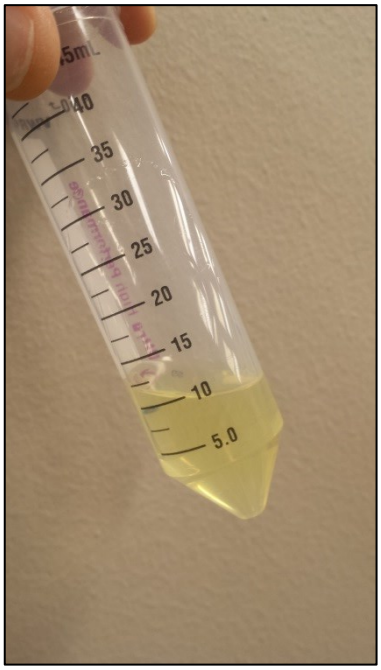
Mixing the soap with water



After adding the red cabbage indicator solution

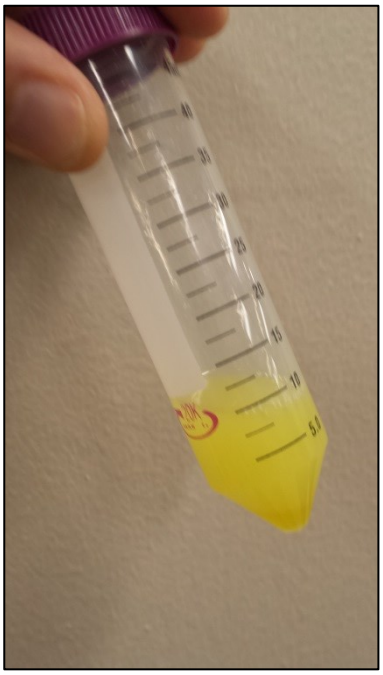
Experiment 12: Crossed Aldol Condensation

Fill a 50-mL test tube with 5 mL of 91% isopropanol. Add 3.5 mL of cinnamaldehyde and 1.0 mL of acetone using two different plastic syringes. Swirl the mixture in the test tube.

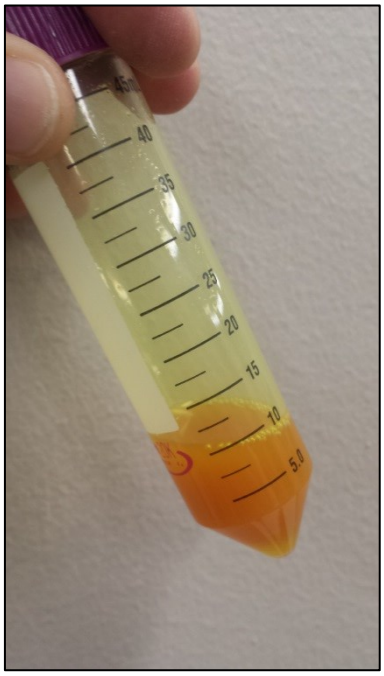


Reaction mixture (no NaOH)

To the solution in the test tube, add 10 drops of 50% NaOH. Cap the test tube and swirl the mixture vigorously for 10 minutes. **Take a few pictures of the test tube over the 10 minute period.**



After 3 minutes



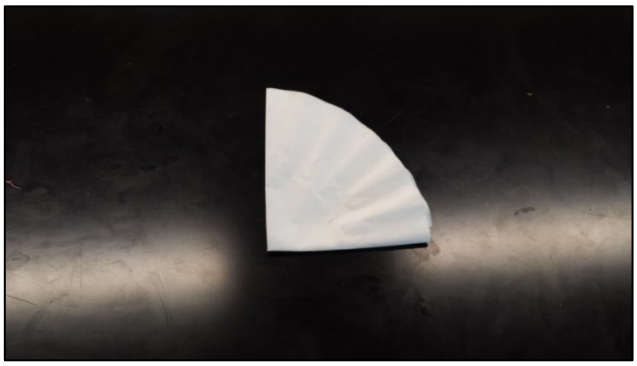
After 5 minutes

After 7 minutes

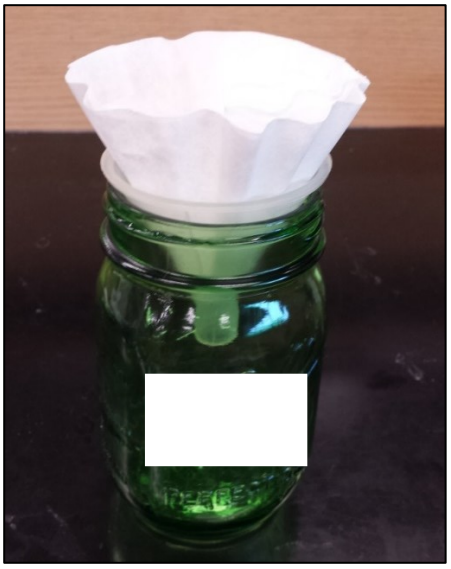
Place the test tube in a bowl of ice and let it sit for 5 minutes.

Cooling in ice bath

While the reaction is cooling, fold the two coffee filters in half (twice) then place it inside the funnel. Put the funnel on top of the jar and filter the cool reaction mixture.



Coffee filter after its folded

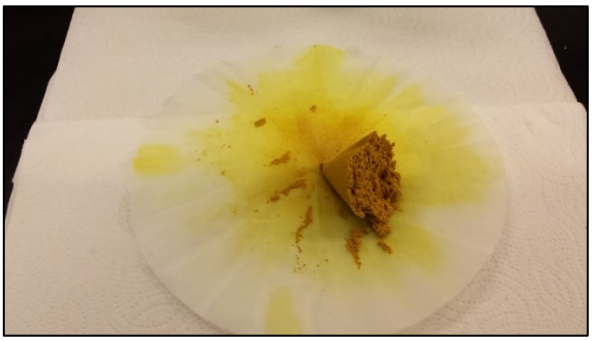


Filtration setup

If the solid sticks to the test tube, pour some ice water into the test tube and use the plastic spatula to break up the solid. Try to transfer as much solid as possible to the funnel for filtration. Wash the solid in the funnel with small amounts of ice water. Allow the mixture to filter until no more liquid is visible dripping from the funnel.

Adding water to mixture **Starting filtration** **Filtration in progress**

Carefully lift the filter paper out of the funnel and place it on a few paper towels. Flatten the solid out on the filter paper and let it air dry for a few hours. **Take a picture of your final product.**



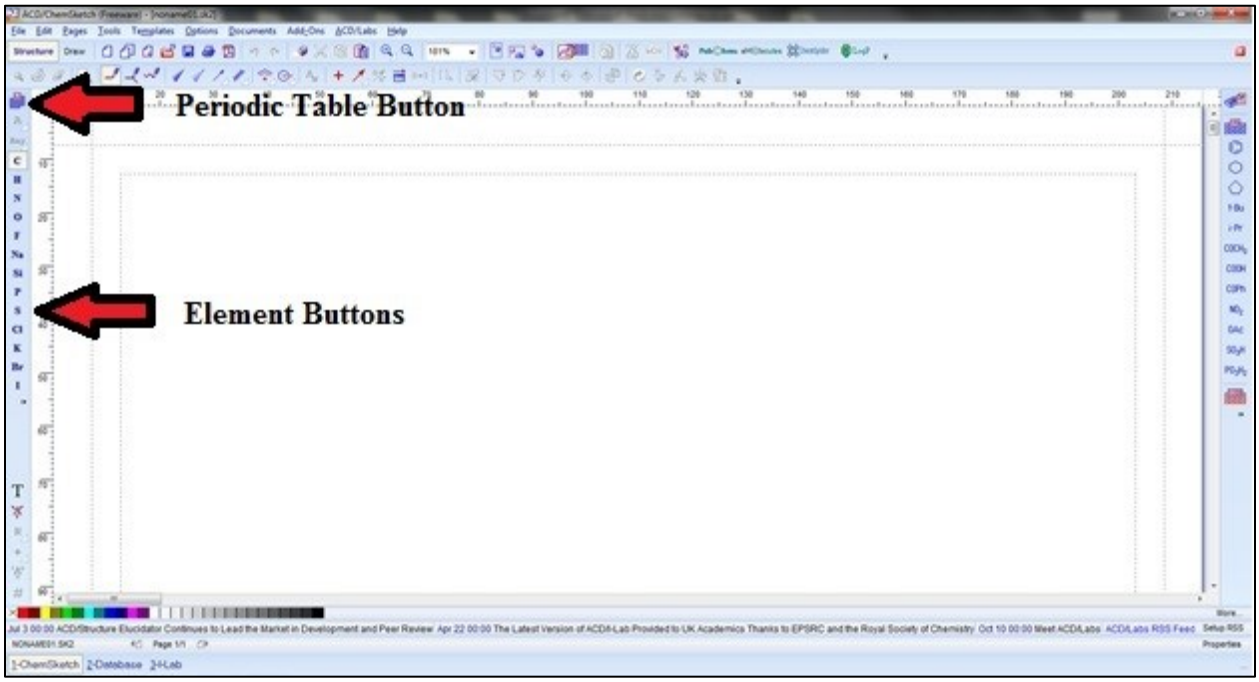
Product still damp **Dried product**

A.3 Student Resources

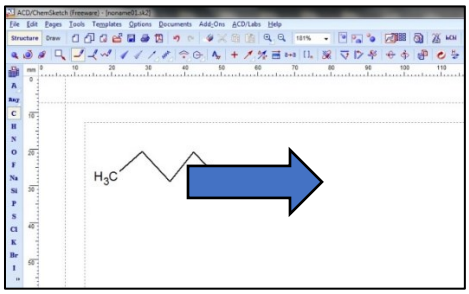
A.3.1 Chemsketch Walkthrough

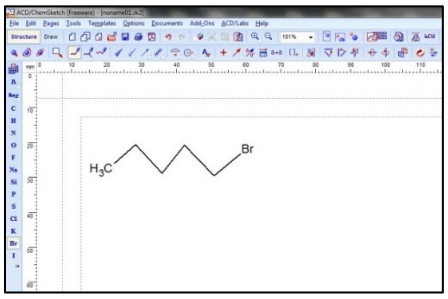
1) After downloading the software on your computer, double-click the Chemsketch icon. As soon as it opens, it will usually show the “Tip of the Day” and a window on how to order the software. Close both windows.

2) You’ll notice down the left hand side of the screen that there are some elements. You can click on those to change what elements that you want to use. If an element is not shown, click the little periodic table icon to find the one that you want.

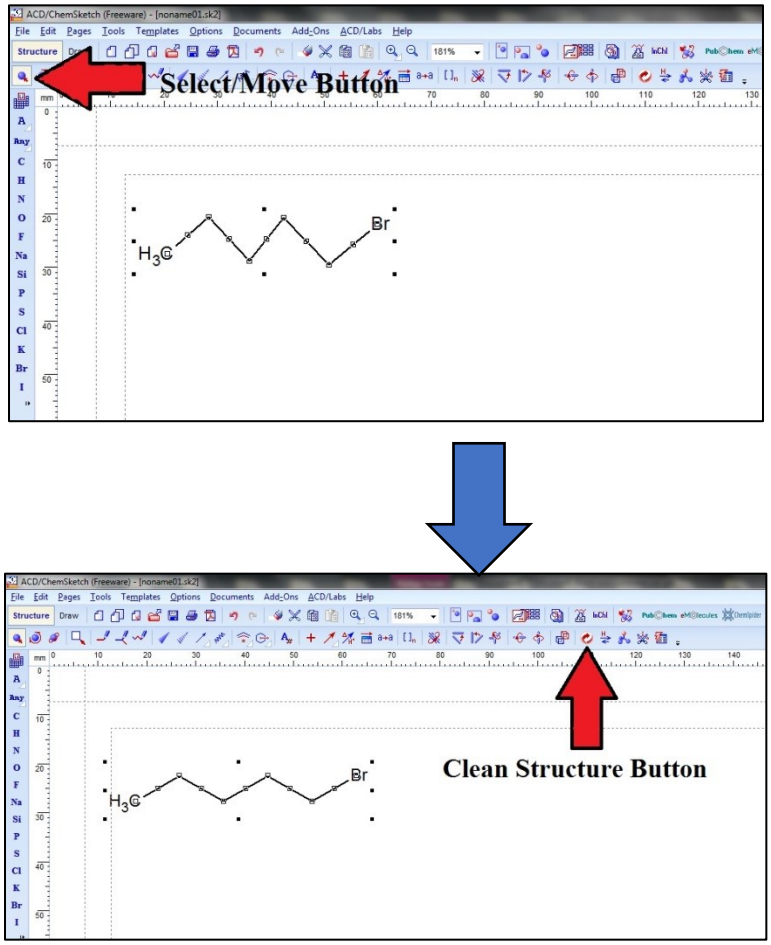


3) Once you select the element that you want to use, click on the white part of the screen and start drawing. For example, if you want to draw 1-bromopentane. Click the carbon icon. Click and drag on the white screen. You'll notice you made a short carbon chain. Click on one of the ends of the carbon chain then drag again. This is how to make longer carbon chains. You'll want to do this a few times until you make pentane (5 carbon chain). To add bromine, click on the bromine icon and click on one of the ends of the carbon chain then drag. Congratulations, you just made 1-bromopentane.

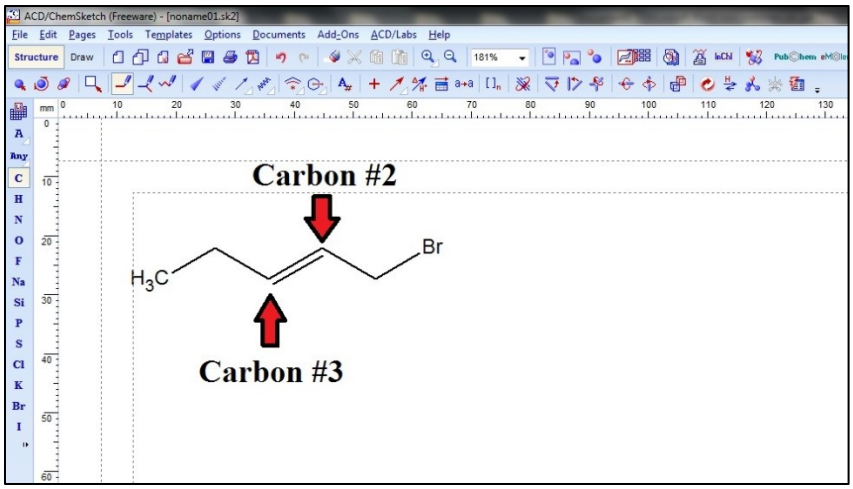




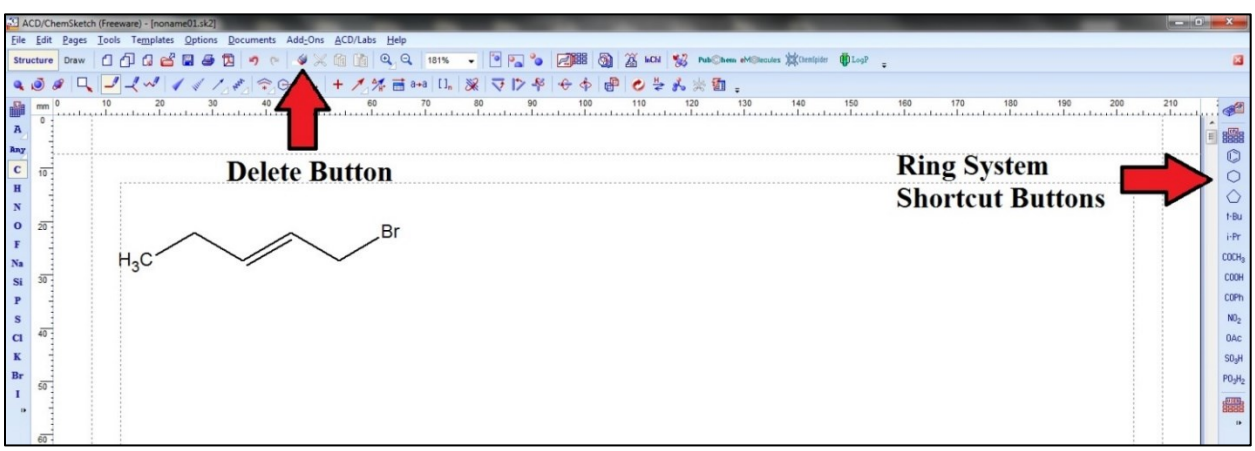
4) To make your structure look pretty, click on the select/move icon. Highlight your structure then click the clean structure icon. This will straighten up the structure making the bond angles and bond lengths uniform.



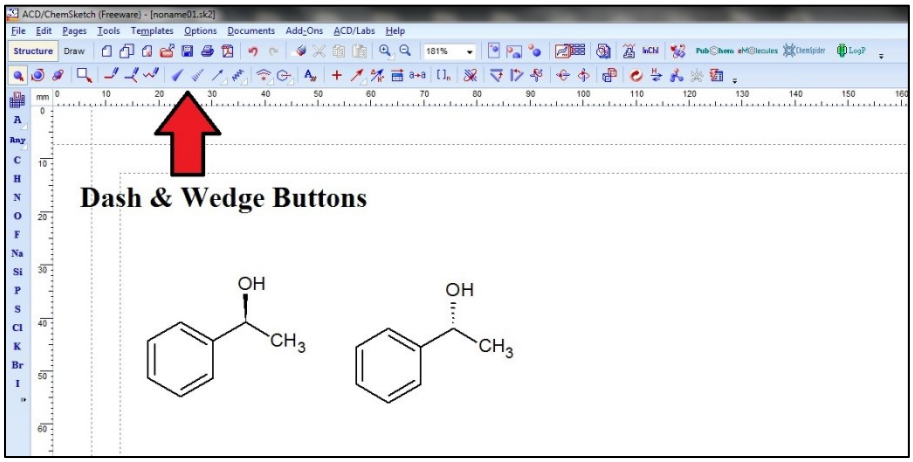
5) If you wanted to make 1-bromo-2-pentene instead, you'll have to add a double bond. To change the bond type (double, triple, etc.), click on the carbon icon then select the carbon where you want to make the double bond, carbon #2, then drag it to carbon #3.



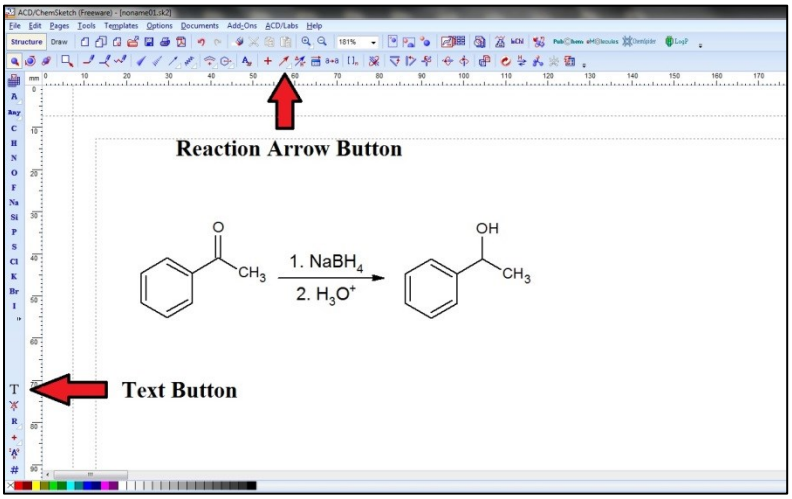
6) To erase parts or all of your structure, click the delete icon (looks like an eraser) then click on the parts of the structure that you want to delete. There are also shortcut icons for drawing ring systems like benzene and cyclohexane; they are located on the right side of the screen.



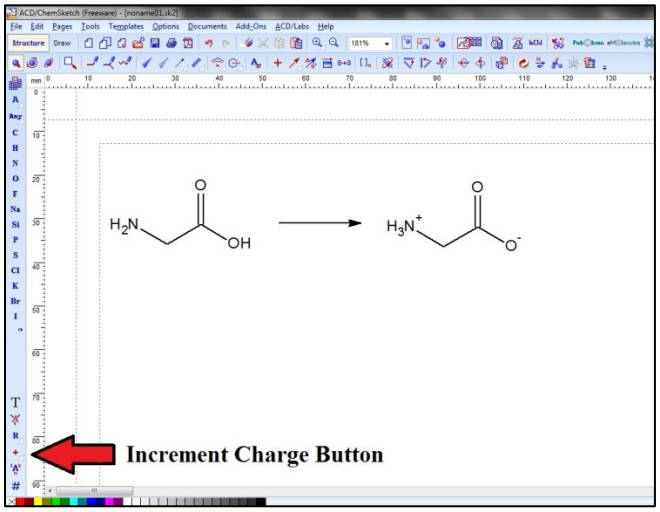
7) To add stereochemistry to a molecule, there are dash and wedge icons. Just select dash or wedge then click on the bond that you want to change.



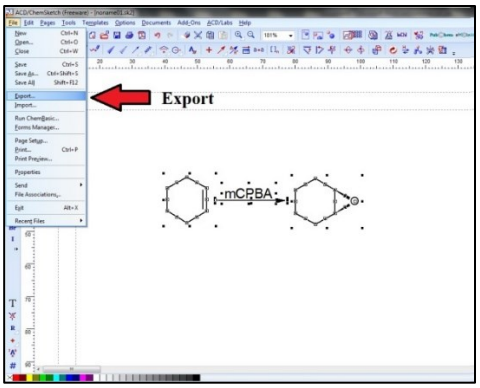
8) To add text the structure, click on the text icon on the left side of the screen. To add a reaction arrow, click on the reaction arrow icon.

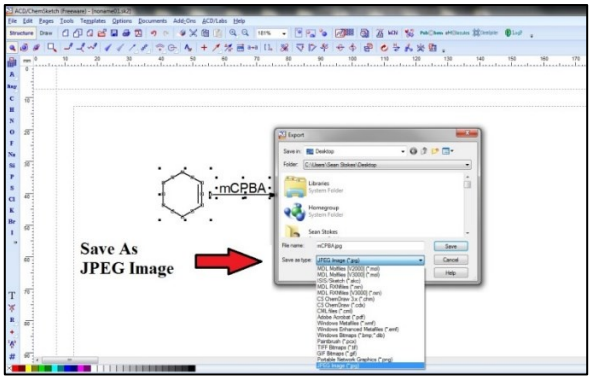


9) To add charges (positive or negative) to atoms, press the increment charge icon. You can select positive or negative by clicking on the small tab on the icon then click on the atom that you want to add the charge to.

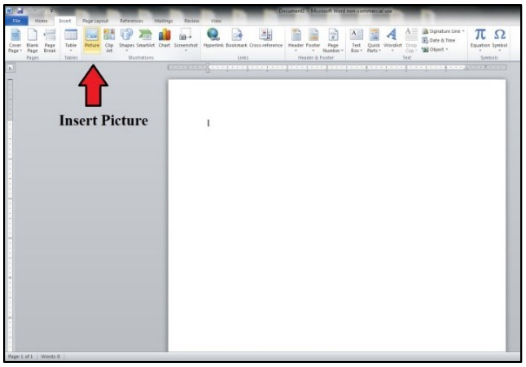


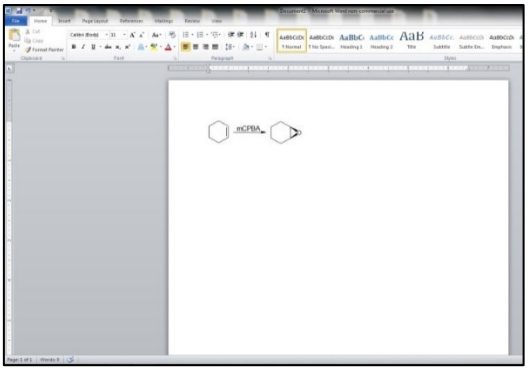
10) To transfer the Chemsketch drawing to a Word document, first select the structure using the select/move icon. Then click on the File tab in the top left corner and click Export. Name the file and save it as a JPEG image to your desktop.



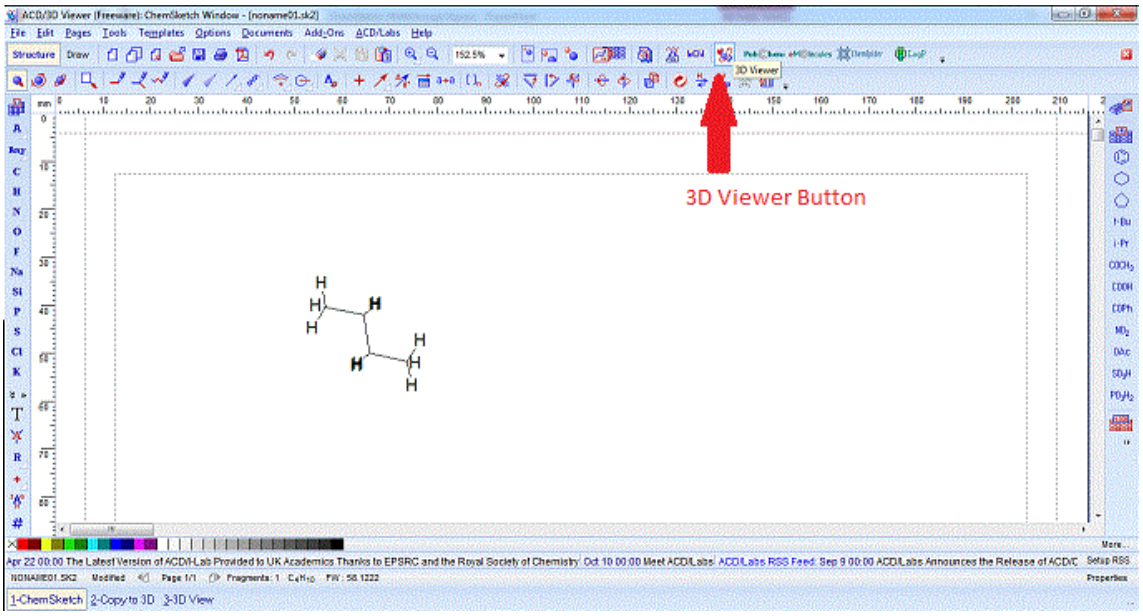


Open the Word document then click the Insert tab. Click picture then find the JPEG image that you saved on the desktop and click insert.

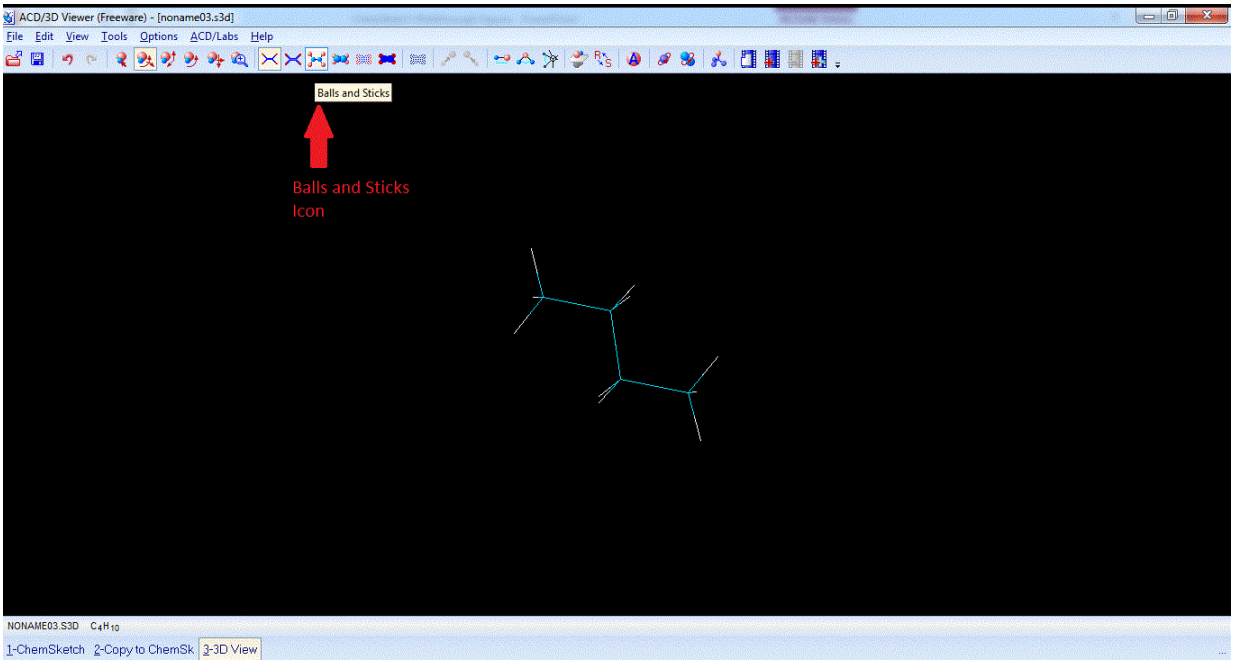




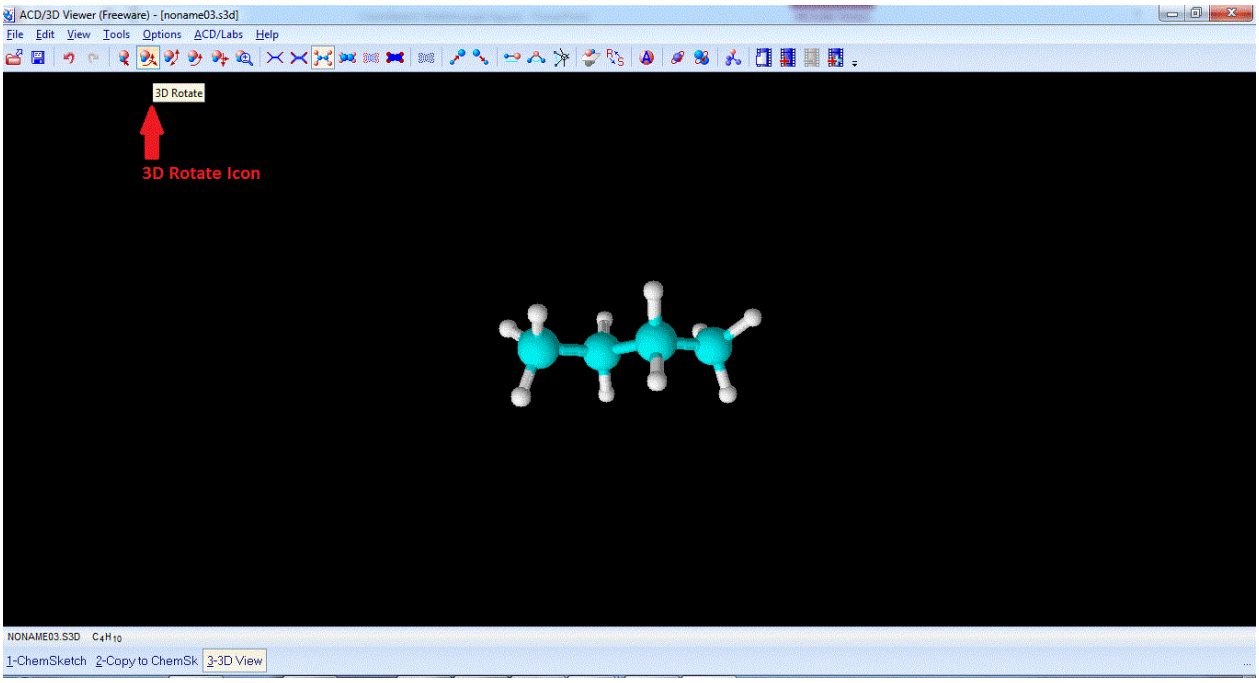
11) To convert your structure to a three-dimensional image (3D), click on the 3D Viewer button on the top-right side of the task bar.



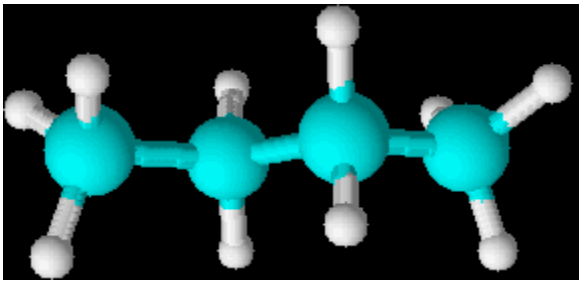
12) Your 3D structure may be showing just as a line drawing like below. To get a pretty image, click on the “Ball and Stick” button.



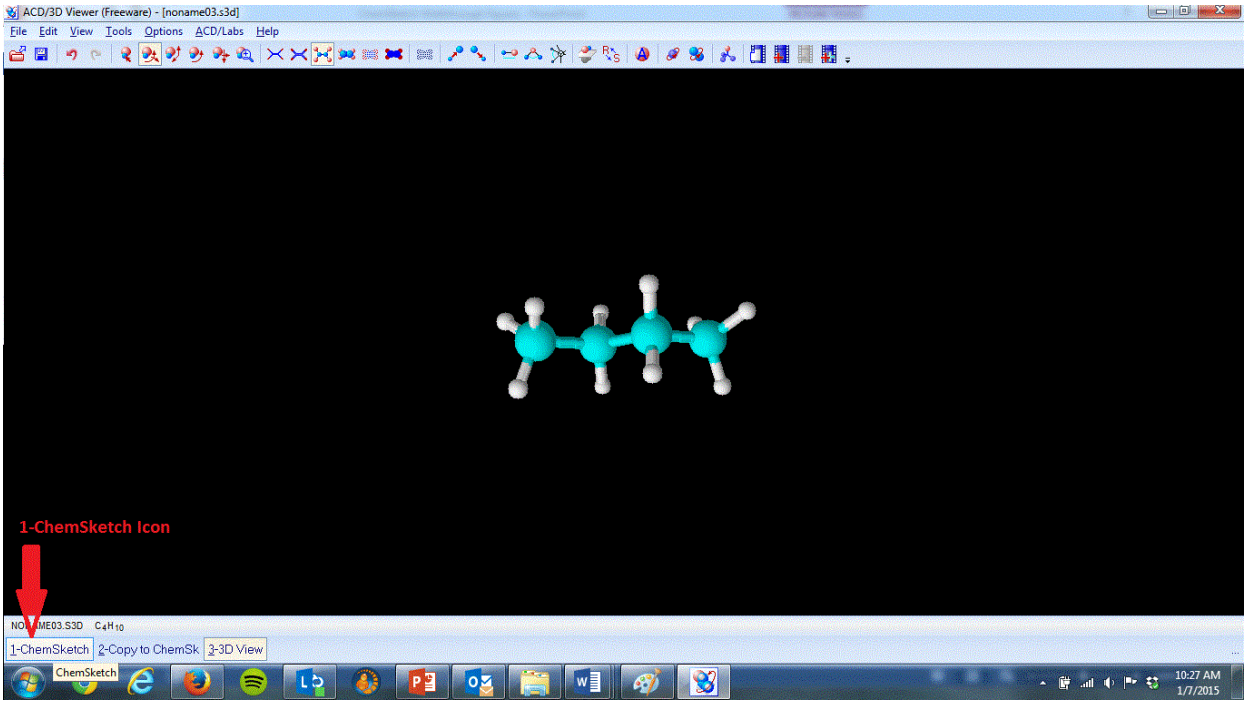
13) If you want to rotate your 3D image, use the “3D Rotate” icon at the top-left part of the screen. If you then click your mouse on the structure and hold down the button, you can rotate the image in any way you choose. To grab the image in a different location, let go of the mouse button and then re-click to grab it differently.



14) To copy your image, click on Edit and then Copy (or do Ctrl-C). Paste into your Lab Report document.



15) To return to the ChemSketch drawing feature, click on “1-ChemSketch” down at the bottom-left of the screen.



A.3.2 CH 2501 Lab Manual

Welcome to CH 2501 Elementary Organic Lab

To get started:

Download and read the syllabus for the course on MyCourses. You are expected to understand the safety and grading policies for the course.

Refer to the Lab Schedule (on the syllabus) for all due dates of assignments.

You MUST follow all the appropriate safety guidelines when doing the experiments from home.

Some of these labs take more than one day to do so PLAN AHEAD. All of the work for this class can be done at home and submitted online.

We will do our best to inform and guide you if you have any questions about the lab experiments or the assignments. Feel free to ask questions of either Dr. Stokes or your TA if you need more assistance.

SAFETY RULES

1. Always wear eye protection. When doing any experiment requiring chemicals, safety goggles must be worn. Prescription eyeglasses are not adequate to provide protection. You are provided with laboratory grade **safety goggles** in your lab kit.

2. **Do not eat food or drink beverages when doing an experiment.** Since some of the chemicals you will be using are household items, they could be easily confused as something that you're eating or drinking.

3. Dress properly during a laboratory activity. Long hair must be tied back, and dangling jewelry and baggy clothing must be secured. **Shoes must completely cover the foot. No sandals.**

4. Use caution when working around a stove or any heat source. Don't burn yourself.

5. Be prepared for your experiment. Read all procedures thoroughly before attempting the experiment. Never fool around while doing an experiment. Some of the chemicals that you will be using are dangerous.

6. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times.

7. Know the locations of all safety equipment around your home like first aid items in case of an emergency.

8. Dispose of all chemical waste properly. Each lab experiment that involves chemicals has a waste disposal section.

9. All chemicals in your lab kit are labeled. Be sure to read them carefully before use. Never return any excess chemicals to a reagent bottle.

10. Keep hands away from face, eyes, mouth, and body while using chemicals or lab equipment. **Wash your hands with soap and water** after performing all experiments.

11. If an accident or serious injury occurs, please notify the instructor or TA immediately.

12. Experiments must be personally monitored at all times. Do not wander off when doing any experiment that requires chemicals.

13. Do not perform any experiments that involve chemicals around small children or pets.

I have read and understand the Laboratory Safety Rules for this course. I do not hold Mississippi State University or its employees responsible for any harm that may come to me or to my property by way of this course.

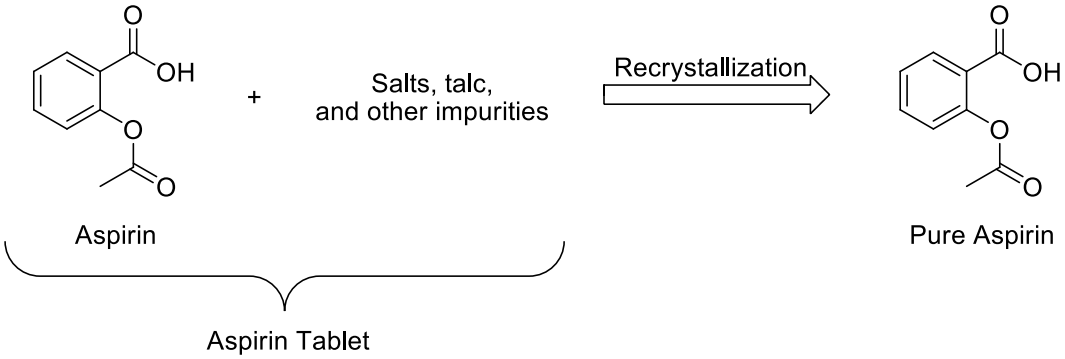
_____	_____	_____	_____
Signature	Print Name	Net ID	Date

Table of Contents

Experiment 1: Recrystallization of Aspirin

Information

Recrystallization is a useful technique that can be used to purify solid organic compounds. This technique involves the solubility of organic solids in various solvents. For recrystallization to work properly, the target compound must dissolve in a solvent when it is hot and not dissolve when it is cold. For example, benzoic acid dissolves very well in hot water but is nearly insoluble in cold water. In this experiment you will isolate pure aspirin from aspirin tablets. Aspirin tablets contain various salts and impurities in addition to aspirin. By using recrystallization, you will be able to remove the impurities and be left with pure aspirin.



Sometimes pairs of solvents can used to produce better results. In this experiment, 91% isopropanol will be used to recrystallize aspirin. The 91% isopropanol contains 91% isopropanol and 9% water so it is really a mixture of two solvents. Most of the impurities will not dissolve in the hot isopropanol solution but the aspirin will. Once the aspirin dissolves, the impurities will be filtered out of the solution. After the solution is cooled, pure aspirin will crystallize out of the isopropanol solution.

Materials

Two 50-mL test tubes
2 tablespoons
4 coffee filters
2 jars
Paper plate
Cooking pot (to heat water)
Plastic funnel
Stove
Refrigerator
Paper towels

Chemicals

Six aspirin tablets
91% isopropanol
Water
Ice

Safety: Make sure to wear your safety goggles when handling any chemicals. You will be using a stove so be careful and don't burn yourself.

Experiment 1: Recrystallization of Aspirin

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. When heating water, be careful not to ( ) yourself.
    b. When handling any chemicals, you must wear your ( ).
2. In recrystallizing aspirin, what are you trying to remove?
3. How will you separate the liquid and solid components during your recrystallization experiment?

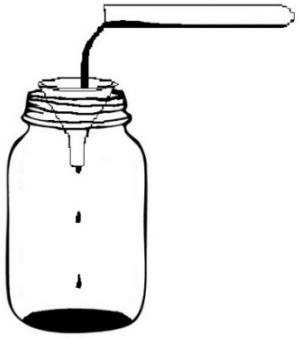
Experiment 1: Recrystallization of Aspirin

Experiment Procedure

Recrystallization: Place six aspirin tablets on a paper plate then crush them with a metal tablespoon (or teaspoon). Carefully transfer the aspirin powder to a 50-mL test tube. Fill the test tube up to the 30-mL mark with 91% isopropanol. Heat up a cooking pot half-full of water on the stove (turn the dial to MED). DO NOT BOIL THE WATER. While the water is heating up, fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of a clean 50-mL test tube. Place this filtration setup (coffee filters, funnel, & test tube) in a jar to keep it upright (see picture below).

Once the pot of water is hot, submerge the bottom half of the aspirin-containing test tube into hot water. You want to heat the aspirin-containing test tube in the hot water. DO NOT CAP THE TEST TUBE. Swirl the test tube for 10-15 minutes in the hot water bath. Some of the solid should dissolve and the solution may appear cloudy. While the solution is still hot, pour it into your filtration setup that you made earlier. You shouldn't see any solid particles in the filtrate. **Take a picture of the filtrate.** Once the filtration is done, add one tablespoon of water to the resulting liquid (filtrate). Let the mixture stand for 10 minutes then put the cap on the test tube. Place the test tube in a jar of ice then put the jar in the refrigerator for 12-24 hours.

After the solution has cooled for 12-24 hours, solid crystals should have appeared at the bottom of the test tube. **Take a picture of the crystals that grew inside the test tube.** Construct another filtration setup similar to the one you made before. Fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of a jar. Filter the aspirin solution using your filtration setup (see picture below). You can use cold water to remove any solid that sticks to the test tube. When it's done filtering, lift the filter paper out and lay it on a few paper towels. Allow the solid to air dry for a few hours. **Take a picture of your dry solid.**



Waste Disposal: All plasticware and glassware should be washed thoroughly with soap and water, dried then placed back in the chemical/equipment kit. The liquid obtained from the filtration can be washed down the sink with copious amounts of water. The solid and filter paper can be thrown away in the trash can. If the solid residue in the test tube doesn't come out, use some 91% isopropanol to remove the solid.

Experiment 1: Recrystallization of Aspirin

Photo Logbook **Name:**

1. Insert a picture of the filtrate after the first filtration step.

2. Insert a picture of the crystals of aspirin that grew inside your test tube.

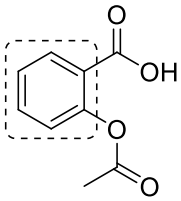
3. Insert a picture of the dried aspirin crystals.

Experiment 1: Recrystallization of Aspirin
Post-Lab Questions **Name:**

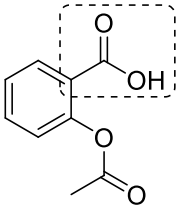
1. What are some of the medicinal uses of aspirin (list at least three)?

2. Aspirin contains many different functional groups. Identify each of the circled functional groups in the molecules of aspirin below.

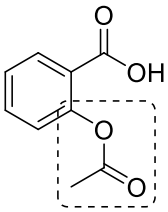
A)



B)



C)



3. What are the following physical properties of aspirin?

   Molar mass:

   Melting point:

   Density:

4. Using Chemsketch, draw the structure of aspirin. Use the arrow function to point to the most acidic hydrogen on the molecule.

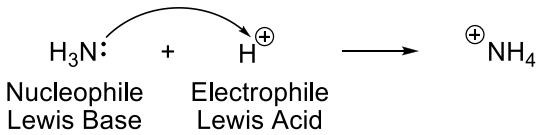
5. Why do you think that aspirin dissolves better in isopropanol than in water?

6. Why was a tablespoon of water added to the filtrate after the first filtration?

Experiment 2: Acids and Bases using Red Cabbage pH Indicator

Acids and bases are enormously important in organic chemistry as many organic reactions involve Lewis acids and bases. In your general chemistry course, you learned about the Bronsted-Lowry concept of acids and bases and this lab is going to explore both definitions.

Lewis Acids and Bases: A ***Lewis acid*** is defined as a chemical entity that can act as an electron-pair acceptor and share the electron pair to form a new bond. Lewis acids tend to have a positive charge or are electron-deficient. Examples of Lewis acids would be carbocations or metal ions. A ***Lewis base*** is defined as a chemical entity that can act as an electron-pair donor. Lewis bases tend to be negatively charged or have extra electrons in the form of double bonds or lone pairs. Examples would be ammonia (NH_3) or fluoride (F^-). When Lewis acids and bases are discussed in organic chemistry, we often use the terms "electrophile" (Lewis acid) and "nucleophile" (Lewis base). Reactions involve a nucleophile sharing electrons with an electrophile as shown in the diagram below.



Bronsted-Lowry Acids and Bases: A ***Bronsted-Lowry acid*** is defined as something that can donate a proton (or H^+). Examples would be hydrochloric acid (HCl) or ammonium (NH_4^+). A Lewis acid doesn't necessarily have to be a Bronsted-Lowry acid. Lewis Bases do also tend to be ***Bronsted-Lowry bases*** (compounds that accept a proton). Examples of B-L bases would be NH_3 or H_2O. With our exploration of acids and bases in this lab, we will be using the Bronsted-Lowry definition to think about acids and bases (losing a proton or gaining a proton). But it is important to understand that when these compounds lose or gain a proton, they are doing reactions similar to the ones you are studying in organic.

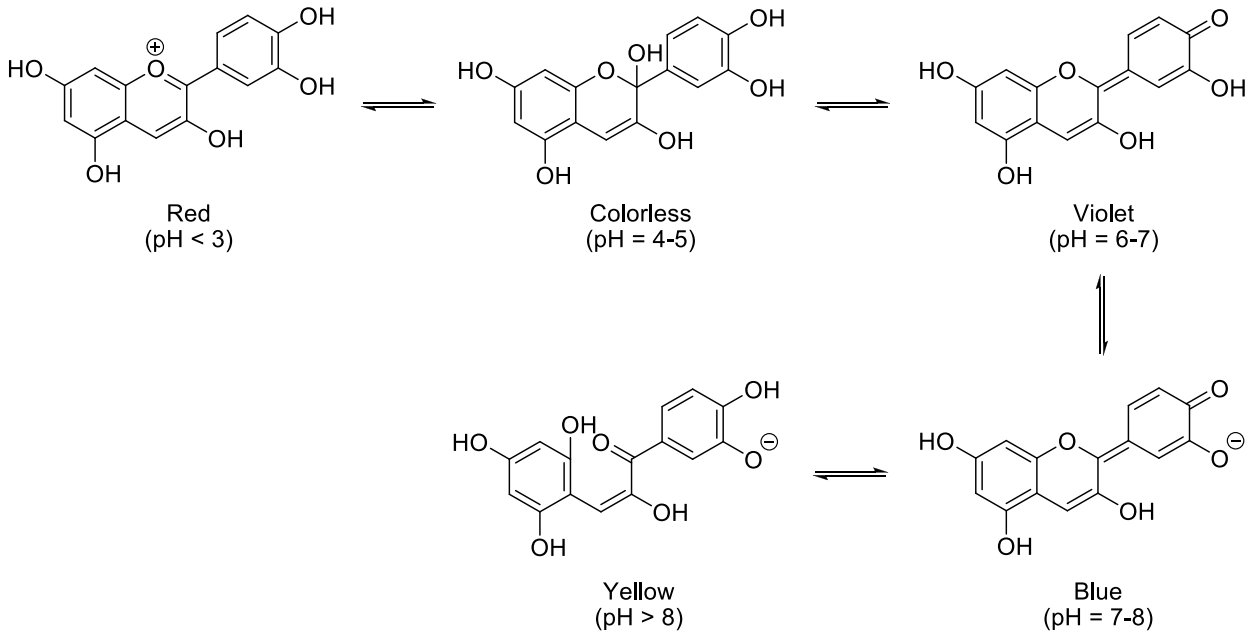
The pH scale: The pH scale is related to the proton concentration in solution. A proton can combine with water to form H_3O^+ known as the hydronium ion and that is how we represent protons in aqueous solutions. The pH of a solution is a way of representing the amount of H_3O^+ and is given by the following equation:

$$pH = -\log [H_3O^+]$$

The pH scale is a logarithmic scale which means that every whole number represents ten times the concentration of H_3O^+.

pH Scale and Relative Ratios of $[H_3O^+]$		
pH scale		Ratio of $[H_3O^+]$ to distilled water
Increasing Acidity	0	10,000,000
	1	1,000,000
	2	100,000
	3	10,000
	4	1,000
	5	100
	6	10
Neutral	7	1
	8	1/10
	9	1/100
	10	1/1000
	11	1/10,000
Increasing Alkalinity	12	1/100,000
	13	1/1,000,000
	14	1/10,000,000

For this lab experiment, you will first create a pH color indicator which will let you estimate the pH of various household chemicals. The color indicator is called anthocyanin (shown below) and is made from red cabbage.



As the pH increases, there are fewer protons in solution. The anthocyanin molecule loses protons from its structure because the phenolic groups are partially acidic. This will cause structural changes in the overall molecule, affecting its amount of conjugation, and causing the molecule to absorb different wavelengths of light. We see the solution as different colors because some of the light wavelengths are being absorbed and removed from the light spectrum. We can use the colors of anthocyanin at different pHs to estimate the pH value of an unknown compound. Values that range from 0-6.9 are termed "acidic" and have high concentrations of H_3O^+; a pH value of 7.0 is defined as "neutral" and values ranging from 7.1-14 are termed "basic" and have low concentrations of H_3O^+. You will use the color table in the worksheet to estimate pH values of common household chemicals (see worksheet).

Materials
One head of red cabbage
A sharp knife
Water
Cooking pot (to use to boil water)
Four clear glass jars or drinking glasses
Measuring cup
Bowl
Refrigerator
Stove

List of possible household chemicals (gather at least 5 items listed below, one from each category)
Vinegar
Lemon juice, orange juice, apple juice or other clear juice
Sprite or clear soft drink like ginger ale or seltzer water
Shampoo or dishwashing soap
Mouthwash or bodywash
Cleaning product like Windex or Fantastik or PineSol
Laundry bleach or liquid plumber
Baking soda or Baking powder (dissolve some in water)
Antacid tablets (crush and dissolve some in water)

Gather an additional 3 items from your household that may have different pH values.
Items can be additional solutions from the list given above or other things you have available. Examples may be things like pickle juice, olive juice, sports drinks, laundry soap, laundry stain pre-treatment, beer or wine, bath salts, canned fruit juice, coffee or aspirin tablets. The clearer a solution is, the easier it is to see any color changes that happens with the pH indicator.

Safety: Make sure to wear your safety goggles when handling any chemicals. You will be using a stove so be careful and don't burn yourself.

Experiment 2: Acids and Bases using Red Cabbage pH Indicator
Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. When using a stove, be careful not to ( ) yourself.
    b. Always wear ( ) when handling chemicals.
2. A Lewis Base can also act as what?
3. Which pH values are defined as "acidic"?

Experiment 2: Acids and Bases using Red Cabbage pH Indicator

Experiment Procedure

Making the pH indicator: Chop about 2 cups of red cabbage into strips (about ¼ of a head) and put in medium sized bowl. Boil 4 cups of water on a stove. Pour the boiling water over the chopped cabbage and let it steep for 10-15 minutes. You want to get a dark, rich color in the water. **Take a picture of your soaking cabbage.** When it is cool, you can scoop the cabbage out so you just have colored extract in the bowl or filter the solution through a few paper towels. Gather up all of your solutions that you want to test. **Take a picture of all the items you gathered for the pH test.**

pH Testing: Pour some of the extract into your four clear glasses. You just need enough to see the color well. Estimate the pH of your extract before adding anything (use the picture in the color table provided in the worksheet to estimate the pH). Pour an equal amount of each of your collected solutions into your clear glasses with the pH extract (one at a time). Estimate the pH of each solution by comparing color to the chart. **Take a picture of all 4 solutions for your photo logbook.** Pour your solutions down the sink (with plenty of water) and rinse the glasses out completely to repeat test with new solutions. Repeat the same procedure for the next 4 solutions. **Take a picture of all 4 solutions for your photo logbook.**

SAVE ½ CUP OF YOUR CABBAGE INDICATOR SOLUTION: Put it in a sealed jar or plastic container and store it in the refrigerator. You will need it for the Soap experiment.

Waste Disposal: All cookware should be washed thoroughly with soap and water. The liquid cabbage extract can be washed down the sink with copious amounts of water (DON'T FORGET TO SAVE SOME). The solids can be thrown away in the trash can.

Experiment 2: Acids and Bases using Red Cabbage pH Indicator

Photo Logbook **Name:**

1. Insert a picture of the cabbage soaking in boiling water.

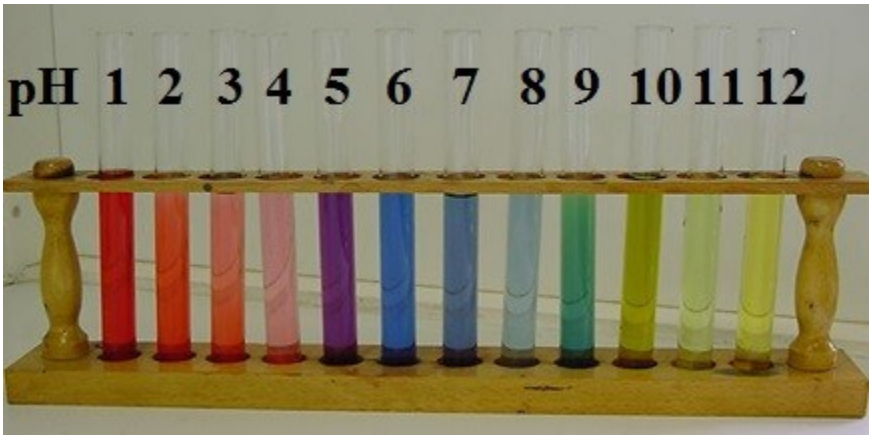
2. Insert a picture of the items you've gathered to test.

3. Insert a picture of the first 4 solutions (Solutions #1-4 on Post-Lab Question #1)

4. Insert a picture of the second 4 solutions (Solutions #5-8 on Post-Lab Question #1)

Experiment 2: Acids and Bases using Red Cabbage pH Indicator

Post-Lab Questions **Name:**



Colors of red cabbage extract at various pH values

1. Complete the table. Estimate pH solutions.

Solution	Color	Estimated pH	Is it acidic, neutral, or basic?
Cabbage extract by itself			
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			

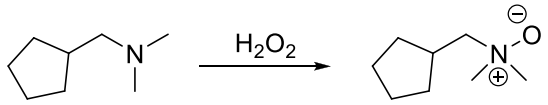
2. Of the items you tested, which one was the strongest acid? Which one was the strongest base?

3. What does a difference of 1 pH unit mean for the concentration of $[H_3O^+]$ in solution? For example, how does a solution with pH of 5 compare to a solution with pH of 6? Which one is the stronger acid?

4. Many soft drinks and juices are acidic because they contain citric acid. Look on the labels of your acidic compounds and list the ones that have citric acid as a component.

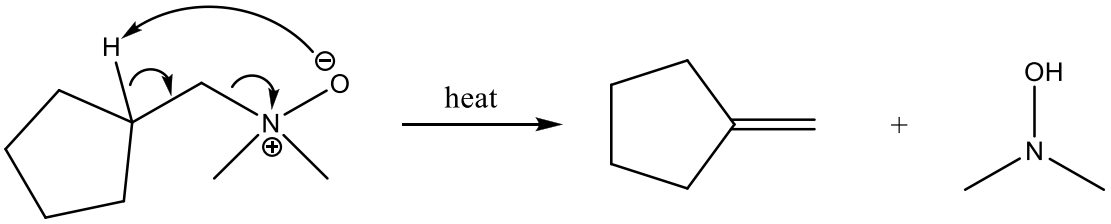
5. Using Chemsketch draw the structure for citric acid. Label or describe the portions of the structure that create acidic solutions. What is this functional group called?

6. Most cleaning products contain amine oxides like the one shown below.



tertiary amine amine oxide

Amine oxides can undergo a Cope reaction (or Cope elimination) to produce an alkene.



In the Cope reaction, does the negatively charged oxygen in the amine oxide…

act as a Bronsted-Lowry acid or base?

_____

act as a Lewis acid or Lewis base?

_____

act as a nucleophile or electrophile?

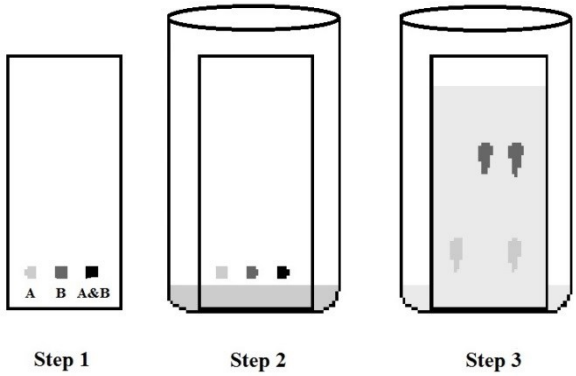
_____

Experiment 3: Separation of Food Dyes by Chromatography

Mixtures can be divided into two major classes – **homogeneous** and **heterogeneous**. A **homogeneous mixture** is composed of two or more pure substances that when mixed together have the physical appearance of uniformity. Salt water is an example of a homogeneous mixture of salt and water. Both salt water and pure water can appear identical – their difference is revealed upon tasting (or evaporating, etc.) the two solutions. A **heterogeneous mixture** contains two or more pure substances, but lacks the uniformity described above. Wet sand is an example of this type of mixture. Close inspection reveals a mixture of a liquid material with a solid material – uniformity, if it exists at all, is confined to small areas or pockets. In addition to these two broad categories, chemists also use the term "**impure substance**" when speaking of a mixture that overwhelmingly contains a single pure substance and small amounts of impurity.

The pure substances that form mixtures can be separated from one another through techniques that exploit both their physical and chemical properties. Physical properties unique to a pure substance are melting point, boiling point, refractive index, and density. In this experiment, we will use a technique called "chromatography" to separate the components of a homogeneous mixture and identify them.

Chromatography is a process in which components of a mixture are exposed to two very different physical environments – a polar phase and a nonpolar phase. In this experiment, the polar phase is the coffee filter (also called the **stationary phase**). The nonpolar phase is typically the solvent that travels up the coffee filter. This solvent is called the **mobile phase** since it travels up the coffee filter through capillary action. Any compound that is nonpolar will prefer to spend time in the nonpolar mobile phase.



Usually, two components in a mixture will not have the same affinity for both phases. The extent of separation depends on each component's time in the mobile phase – the longer a component is in the mobile phase, the farther it will travel along the plate. In the picture shown above, pure A, pure B and a mixture of A & B are spotted at the bottom of the stationary phase in Step 1. The mobile phase is allowed to pass through the components in Step 2 and carry them up the stationary phase. The stationary phase is withdrawn in Step 3 and the positions of A and B are located – note the separation between A and B that occurred in the third lane. The compounds separate because of the "polarity" of the stationary phase and the mobile phase; the stationary phase is polar in this case, and the mobile phase is nonpolar.

Interesting side note: Chromatography is a very wide-spread technique that is used in all branches of chemistry and biochemistry. It is used to purify all the proteins and organic samples that scientists study. A specialized application of chromatography is used for "DNA fingerprinting" that allows forensic chemists to match the DNA from a crime scene to that of a suspected perpetrator.

In today's experiment, we will perform several separations involving food dyes. Standard dyes will be spotted on a coffee filter and compared with unknown solutions that contain combinations of the dyes. Comparison of the R_f values will you to identify and differentiate each dye based on their separations. Shown below is a table of the pigments commonly used in food dyes. The R_f value (which stands for **retention factor**) is a number that corresponds to how far a component travels versus how far the solvent travels.

$$R_f = \frac{distance\ travelled\ by\ component}{distance\ travelled\ by\ solvent\ front}$$

The R_f value can be calculated either from the most intense point of the component spot OR from the first edge of the component spot. You can compare the R_f values either way – for the purposes of this lab, you just need to be consistent. It is important that you mark the solvent front right after the chromatogram develops as the solvent will evaporate quickly and you'll lose where the front is.

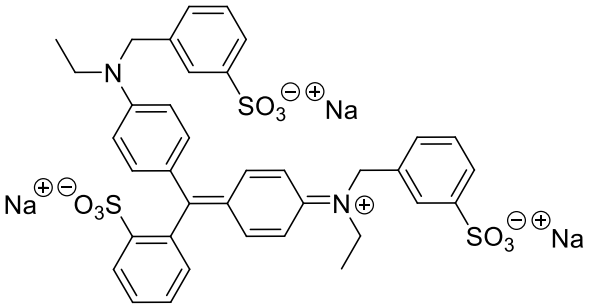
We typically use food dyes to give intense and vibrant color to foods. Some of the reasons this may be desirable is to:

- Offset color loss in the food due to exposure to air, heat or other storage conditions.

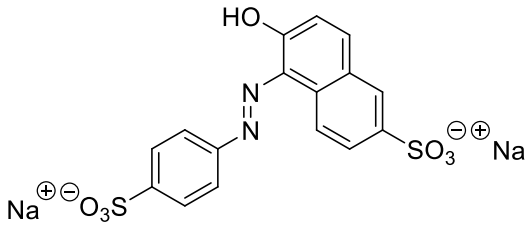
- Provide a color identity to foods that would otherwise be colorless, such as ice cream.
- To correct natural variations in color so samples appear consistent.
- Act as a light filter to potentially protect flavors and vitamins that can be degraded by light.
- Provide an appealing array of foods that meet consumer demands.

Food dyes fall into two categories: either “natural” or “synthetic”. Natural pigments are obtained from animal, vegetable or mineral sources and are not required to be certified by the FDA for use. Natural colors tend to be less stable than synthetic dyes and often fade in color or intensity over time. Synthetic dyes must be approved by the Food and Drug administration for human consumption. The synthetic dyes all have common characteristics, namely:

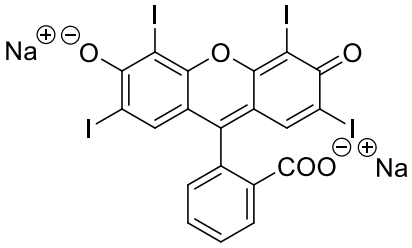
- The dyes all contain numerous double and triple bonds. It is this bond structure that gives them intense colors in visible light. These dyes often contain a –N=N– group as well and so are designated as “azo” compounds.
- While the dyes are mostly carbon and hydrogen, all of them contain some ionic components such as the $-SO_3^-Na^+$ or $-CO_2^-Na^+$ portions. These anionic regions make the dyes water soluble.



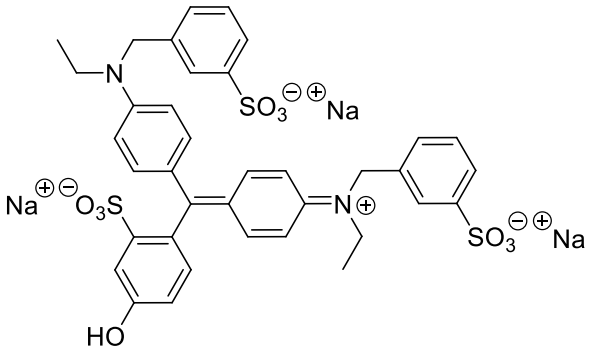
Brilliant Blue FCF (**Blue 1**)
The most common blue dye.



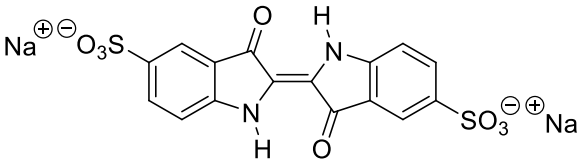
Sunset Yellow FCF (**Yellow 6**)
Known to cause mild allergic reactions.



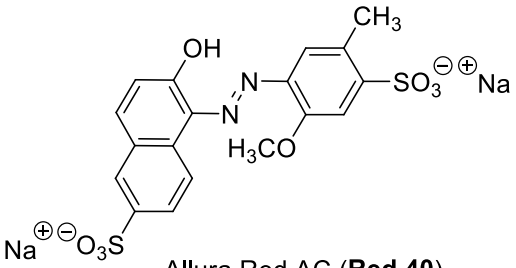
Erythrosine (**Red 3**)
The most common red dye.



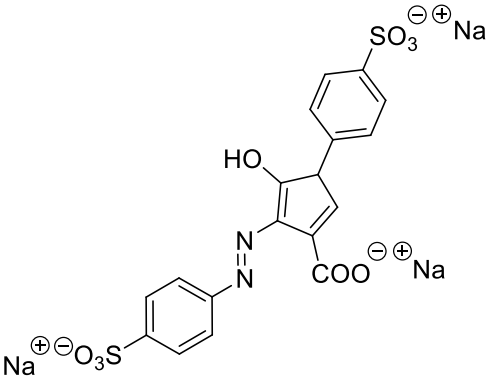
Fast Green FCF (**Green 3**)
One of the least used dyes.



Indigotine (**Blue 2)**
Light sensitive & decomposes over time



Allura Red AC (**Red 40**)



Tartrazine (**Yellow 5**)
Known to cause mild allergic reactions

Why is the dye colored? Dyes are organic molecules that selectively absorb wavelengths of light in the visible range of the spectrum. When an object absorbs a specific wavelength, we see all of the others that are left over and the object appears to have a "color". If orange light is filtered out of "white" light, then we see the object as blue-green, which is the complementary color for orange. Whenever a wavelength is absorbed, we see the color as the complementary color to that wavelength.

Complementary Colors

Color Absorbed	Wavelength (nm)	Color Observed
Red	647-700	Green
Orange	585-647	Cyan (green-blue)
Yellow	570-585	Blue
Green	491-570	Red
Blue	424-491	Yellow
Violet	400-424	Yellow-Green

What determines the wavelength a dye will absorb? The color in azo dyes is due to the large systems of conjugated double bonds that can be delocalized. These delocalized electron systems absorb specific wavelengths of light. In general, the more conjugation you have in the system the lower the energy of light that the compound absorbs. Low energy light is associated with longer wavelengths which are red; so dyes with higher conjugation absorb red and appear green (red's complementary color).

Materials

Coffee filters
Two jars
Paper clip or toothpick
Pencil
Ruler

Three small plastic cups or glasses
Teaspoon
Paper plate
Plastic pipet
Measuring cup

Chemicals

Standard food color dyes (4 colors)
Table salt (sodium chloride)
91% isopropanol
Water
M&Ms or Skittles (3 candies, 3 different colors)

Safety: Make sure to wear your safety goggles when handling any chemicals. The food coloring may stain your hands or clothes so you may want to wear gloves and an apron when doing this experiment.

Experiment 3: Separation of Food Dyes by Chromatography

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

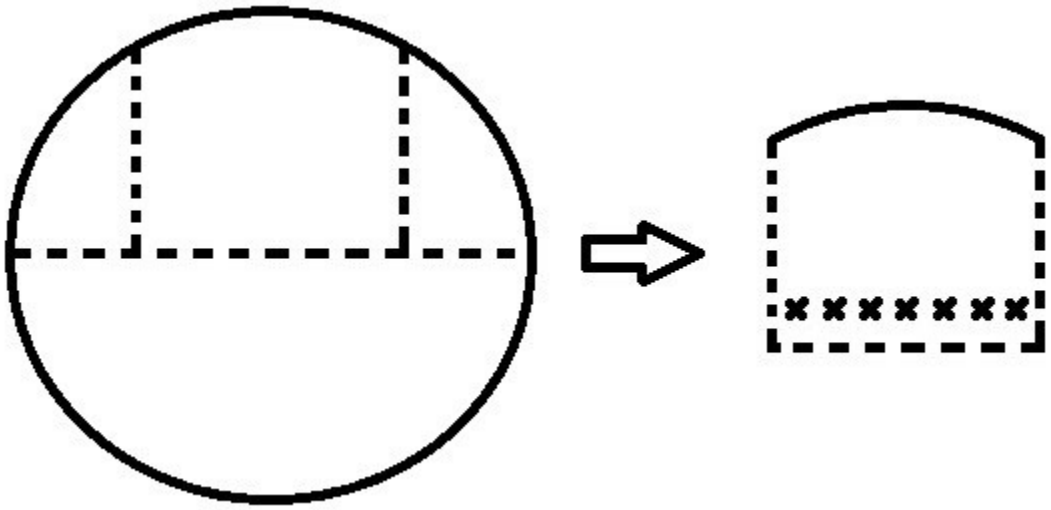
Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. When handling any chemicals, you must wear your ( ).
    b. Food coloring may stain your clothes or hands, so you may want to wear ( ).
2. How can the R_f value be used to identify unknown dyes from your candies?
3. What about these dye structures make them appear colored to us?

Experiment 3: Separation of Food Dyes by Chromatography

Experiment Procedure

Preparing the Coffee Filters: Gently flatten out a coffee filter on the countertop. Cut the filter in half then cut the rounded edges off the sides. Draw seven light "X" marks on the bottom cut edge with a PENCIL. You should place your marks about 1.0 cm from the bottom edge of the filter paper (see picture below).



Preparing the M&Ms or Skittles for Chromatography: Pull out 3 colors of M&Ms or Skittles and place each color in its own plastic cup. Add 6 drops of 91% isopropanol and 6 drops of water to each cup with a plastic pipet. Soak each candy for about 15 minutes. Swirl the mixture occasionally to get the maximum amount of dye off the candy. You want to get a rich concentrated color in your isopropanol solution. **Take a picture of the M&Ms or Skittles soaking the isopropanol solution.**

Preparing Chromatography Chamber: Mix 1.5 tsp of table salt (NaCl) in a ½ cup of water in a clean jar. Make sure all the salt dissolves before use. Pour some of the salt solution onto a second jar; you barely want to coat the bottom of the jar with the salt solution. This second jar will be your chromatography "chamber".

Preparing Food Dyes for Chromatography: Squeeze out a small drop of the 4 food dyes onto your paper plate. Check your box of food dyes to identify the dye colors included in your box. The most common ones are Red #40, Yellow #5 and Blue #1. Write down your dye colors on your worksheet.

Running the Dye Samples: Dip a clean toothpick or unfolded paper clip into each dye solution (the 4 four food dyes and 3 candy solutions) and dot each gently onto one of the "X" marks of your cut coffee filter. Place one color on to each "X" spot. You want to dot your sample multiple times (2-3 times) while each time having the spot remain as small as possible. Let the spot dry in between each application so that the spot can stay as small as possible. Wipe your toothpick clean (or get a new one) after each dye so you don't contaminate one spot with another. After all the colors are on the coffee filter, place the coffee filter in your chromatography chamber. Be careful that the solvent stays BELOW your "X" marks where your spots are marked and also make sure the cut edge of your coffee filter is down in the liquid. Allow your coffee filter to sit UNDISTURBED while the salt water moves up the sheet. Watch and remove the coffee filter from solution when the solvent line ~ 1 cm from the top edge of the filter. Mark the edge of the solvent lightly with a pencil all the way across the filter. The solvent is very volatile and will begin to evaporate soon, so do not delay. Wait for your coffee filter to dry and then gently flatten it out on the counter. Lightly circle all of the dye spots present on your plates with a pencil. Measure the R_f values for each of the different dye spots and record your data on your worksheet. When measuring R_f values, measure from the "X" mark to the leading edge of the dye spot then divide by the distance from the "X" mark to solvent front. Identify your unknown dyes. Match your unknown dye spots to the 4 known dyes from the food coloring. If they have the same R_f value, then they are a match. If you have a spot with an R_f value that does not match, list it as an unknown. **Take a picture of the coffee filter after running the dyes in the chromatography chamber.**

Waste Disposal: The jars and any other glassware should be washed thoroughly with soap and water. The used M&Ms or Skittles can be thrown away in the trash can. Any excess salt solution can be pour down the sink with copious amounts of water.

Experiment 3: Separation of Food Dyes by Chromatography

Photo Logbook **Name:**

1. Insert a picture of the M&Ms or Skittles soaking the isopropanol solution.

2. Insert a picture of the coffee filter after running the dyes in the chromatography chamber.

Experiment 3: Separation of Food Dyes by Chromatography

Post-Lab Questions **Name:**

1. Fill out the information below regarding each dye that you ran in your chromatography experiment. Note: some unknowns may have more than one color; be sure to list all of them. If you cannot identify the dye present in your M&Ms or Skittles, then report it as an "unknown dye".

Spot Description	**Colors Present**	**Distance Solvent traveled (cm)**	**Distance Dye traveled (cm)**	**Calculated R_f value for each dye**	**Identity of Dye**
Red Dye #					
Yellow Dye #					
Blue Dye #					

2. Can you identify any of your unknown spots as corresponding to a specific known dye? How can you tell?

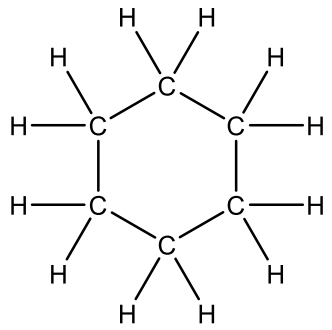
3. What is the stationary phase and mobile phase in this experiment?

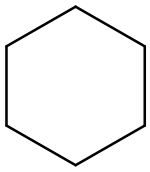
Experiment 4: Molecular Modeling of Cyclohexane and Substituted Isomers

This lab experiment will use ChemSketch to model cyclohexane and related substituted isomers in order to emphasize the structure of the compounds. Read the ChemSketch Download instructions and tutorial provided for you prior to this Molecular Modeling experiment.

Cyclohexane can be represented as a six-sided carbon ring with hydrogens attached.

Figure 1. Flat, 2-Dimensional representations of cyclohexane.



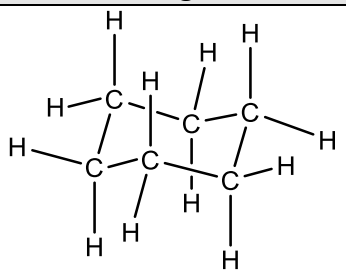


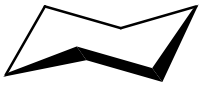
1a: Lewis structure for cyclohexane

1b: Skeletal structure for cyclohexane

But cyclohexane doesn't really lay flat. Instead it forms a kinked structure that we refer to as a chair conformation. This kinked structure allows all of the bond angles to have less stress on them.

Figure 2. Chair conformation for cyclohexane.





2a: Lewis structure of cyclohexane chair conformation

2b: Skeletal structure of chair cyclohexane

Some hydrogens extend up and down in the chair conformation. These are known as “axial” hydrogens. Larger groups have a hard time existing in these axial positions as the groups are closer to the other axial groups. We refer to this as steric hindrance. Six of the hydrogens in cyclohexane chair are extending out toward the sides. These hydrogens are known as “equatorial” hydrogens as they are around the equator or middle of the structure. Equatorial groups have more space as they extend out and away from the other atoms in the structure. Larger groups get priority in the equatorial positions to minimize the steric hindrance.

Experiment 4: Molecular Modeling of Cyclohexane and Substituted Isomers

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. What is steric hindrance?
2. List the two categories of hydrogens found in cyclohexane.
3. Which location in question #2 is more stable for large groups and why?

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Experiment Procedure

The Chair (Cyclohexane): Go into ChemSketch and construct a model of cyclohexane by joining six carbons in a ring and then adding two hydrogens to each carbon (see Tutorial provided for help getting started). Go to Tools and select 3-D structure optimization. If it asks you about removing hydrogens, hit "No". Click on 3-D rotate button on the ToolBar (3rd button from left; looks like a planet with a ring around it). Rotate the model until you have something that looks close to the chair structure shown in Figure 2 above. You want to have one carbon heading up like the back of a chair, and one carbon facing down like the legs of a chair. Select your whole molecule by mouse-clicking outside of your molecule and opening up a box around your molecule. After you draw the box, it will select all the atoms and put little boxes on them. Go up to the top Toolbar and click on "3-D Viewer" – it is toward the right side and looks like a ball-and-stick model with an arrow circling it. Your ChemSketch will now open the 3-D viewer screen and display your molecule in 3 dimensions. Change your viewer to Ball-and-Stick version by clicking on the button halfway across that looks like a ball-and-stick model. Rotate your 3-D model until you again see the chair conformation. Under "Edit", click Copy. Paste the image of your model into your photo logbook page.

Methylcyclohexane: Go to the bottom left corner of your 3-D screen and click on 1-ChemSketch. This will take you back to the 2-dimensional drawing program. Click on a C (carbon) on the left side and click on any H (hydrogen) on your model. The H will be replaced by a $-CH_3$ (methyl) group. Select your model by drawing a box around it with the mouse. Under Tools, hit "3-D Optimization" and answer "no" when it asks if you want to remove the hydrogens to optimize. Your model will now show in the drawing program as a 2-D methylcyclohexane. Select your model and hit the button at bottom left corner of screen that says "Copy to 3-D". Switch to the 3-D viewer to view your new model. Under Edit, hit Copy and copy your model image into your photo logbook.

***trans*-1,2-Dimethylcyclohexane:** Go back to the 2-D ChemSketch viewer (bottom left button). Add in another C adjacent to your methyl group. You want to have one of them pointing up, and the other one pointing down so they are *trans* to each other, or on opposite sides. Select your model by drawing a box around it with the mouse. Under Tools, hit "3-D Optimization" and answer "no" when it asks if you want to remove the hydrogens to optimize. Your model will now show in the drawing program as a 2-D trans-dimethylcyclohexane. Select your model and hit the button at bottom left corner of screen that says "Copy to 3-D". Copy your 3-D model and input into your photo logbook.

***cis*-1,2-Dimethylcyclohexane:** Repeat all of the steps and create the cis version of 1,2-dimethylcyclohexane. Copy the 3-D image for your photo logbook.

1,3- and 1,4-Dimethylcyclohexane: Now make a model of *cis*-1,3-dimethylcyclohexane. Repeat for *trans*-1,3-dimethylcyclohexane, *cis*-1,4-dimethylcyclohexane, and *trans*-1,4-dimethylcyclohexane. Copy all of your images into your photo logbook.

Bromocyclohexanes: Indicate the preferred position (axial or equatorial) of the bromo substituent in each of the following. The *tert*-butyl group is so large that it will always be in the equatorial position. Copy and paste at least one of these isomers into your photo logbook.

a. *trans*-1-bromo-4-*tert*-butylcyclohexane
b. *cis*-1-bromo-3-*tert*-butylcyclohexane
c. *trans*-1-bromo-3-*tert*-butylcyclohexane
d. *cis*-1-bromo-2-*tert*-butylcyclohexane
e. *trans*-1-bromo-2-*tert*-butylcyclohexane
f. 1-bromo-1-*tert*-butylcyclohexane

Experiment 4: Molecular Modeling of Cyclohexane and Substituted Isomers

Photo Logbook **Name:**

1. Insert pictures of each of the following 3-D structures.
   a. Cyclohexane:

   b. Methylcyclohexane:

   c. *trans*-1,2-dimethylcyclohexane:

   d. *cis*-1,2-dimethylcyclohexane:

   e. *trans*-1,3-dimethylcyclohexane:

   f. *cis*-1,3-dimethylcyclohexane

   g. *trans*-1,4-dimethylcyclohexane:

   h. *cis*-1,4-dimethylcyclohexane

   i. Bromo-isomer:

Experiment 4: Molecular Modeling of Cyclohexane and Substituted Isomers

Post-lab Questions **Name:**

Cyclohexane Chair conformation:

1. Arrange your chair model so it looks like a chair…you will find that some hydrogens are sticking straight up and some are sticking straight down; these are known as "axial" hydrogens.

   a. How many hydrogens are sticking straight up?

   b. How many hydrogens are sticking straight down?

   c. How many hydrogens are sticking out to the sides of the model? (you may need to rotate it to count them all). These are known as "equatorial" hydrogens because they are going around the middle, or "equator"

Methylcyclohexane:

2. Is your methyl group in an axial position or an equatorial position?

3. Why might the methyl group be more stable in that spot? Explain your answer.

***trans*-1,2-Dimethylcyclohexane:**

4. *trans*-1,2-dimethylcyclohexane is most stable when the two methyl groups are in which position: axial or equatorial?

5. Why are the methyl groups more stable in that spot? Explain your answer.

***cis*-1,2-Dimethylcyclohexane:**

6. Look carefully at your structure and count the axial and equatorial hydrogens. What are the locations of the two methyl groups: axial or equatorial?

7. What might cause steric hindrance in this molecule?

All Dimethyl Isomers:

8. Of all these dimethyl isomers (including the cis and trans-1,2-dimethyl isomers) which one would be most stable? Why?

9. Which one would be least stable? Why?

Bromocylohexanes:

10. Indicate the preferred position (axial or equatorial) of the bromo substituent in each of the following.

    a. *trans*-1-bromo-4-*tert*-butylcyclohexane

    b. *cis*-1-bromo-3-*tert*-butylcyclohexane

    c. *trans*-1-bromo-3-*tert*-butylcyclohexane

    d. *cis*-1-bromo-2-*tert*-butylcyclohexane

    e. *trans*-1-bromo-2-*tert*-butylcyclohexane

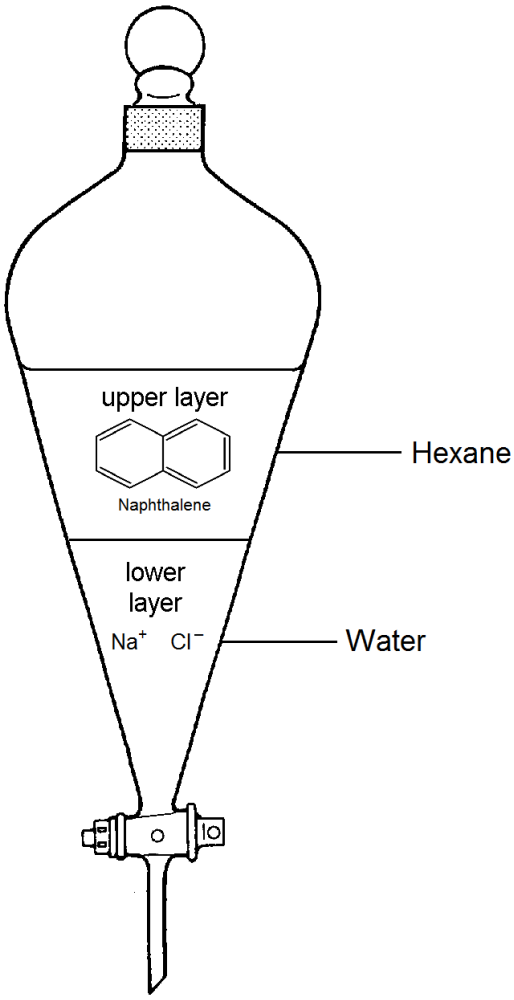
    f. 1-bromo-1-*tert*-butylcyclohexane

Conclusions:

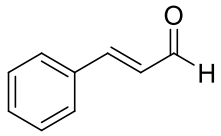
11. Create a set of rules that explains how groups arrange in cyclohexane. Be sure to address group size and the number of groups attached with your rule.

Experiment 5: Extraction

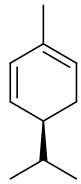
Extraction is a commonly utilized technique in organic chemistry. It can be used to separate different types of molecules from one another. Simply put, it functions on the principle of "like dissolves like." Alike compounds will tend to stick together (polar with polar and non-polar with non-polar). If you had a mixture of two different types of compounds, you can use different solvents to separate the mixture. For example, if you had a mixture of NaCl and naphthalene (both are colorless solids) you could use a solvent like hexane to extract the naphthalene from the mixture and water to extract the NaCl. Naphthalene is a non-polar hydrocarbon like hexane so they should mix readily. Water and NaCl will mix based on the same principle (like dissolves like) except they are both polar.



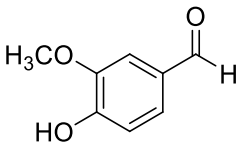
Plant essential oils and extracts are mixtures of organic compounds that have been isolated from raw plant material like leaves, roots or bark. Some of these extracts contain volatile components that have a characteristic odor or "essence". In this experiment you will created two plant extracts (one from lemon peel and the other from ground cloves). These two plant extracts will be tested for the presence of alkenes using a solution of $KMnO_4$.



cinnamaldehyde
from cinnamon bark

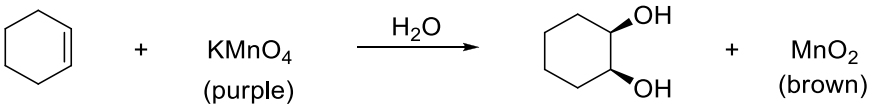


(*S*)-alpha-phellandrene
from dill leaves



vanillin
from vanilla beans

The potassium permanganate ($KMnO_4$) has a very distinct dark purple color. If $KMnO_4$ is added to a solution containing an alkene, the alkene will be oxidized to form a diol. As the reaction proceeds, a brown solid will precipitate from solution. This brown solid is manganese(IV) oxide. If $KMnO_4$ is added to a solution that doesn't contain an alkene, the solution will remain purple.



When $KMnO_4$ reacts with an alkene, the diol that is produced has *syn stereochemistry*. Notice: the two alcohol functional groups in the product on the same face (or side).

Materials

Four jars with metal lids
Four coffee filters
Plastic funnel
Two 15-mL test tubes
A few paper towels
One 50-mL test tube
Two plastic pipets
A few rubber bands

Chemicals

One fresh lemon
Two tablespoons of ground cloves (in baggie)
91% isopropanol
$KMnO_4$ solution (in a dropper bottle)

Safety: Make sure to wear your safety goggles when handling any chemicals. The $KMnO_4$ solution will stain both clothes and skin.

Experiment 5: Extraction

Pre-lab Exercise **Name:**

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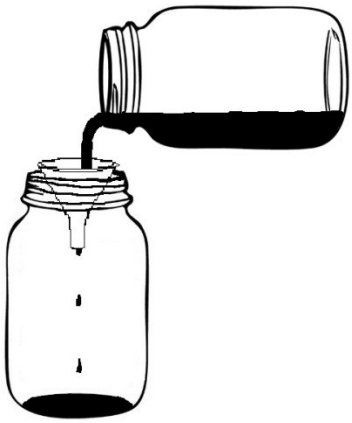
Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. When handling any chemicals, you must wear your ( ).
    b. What solution that you are working with will stain your clothes and skin?
2. How does extraction work to separate out a mixture of compounds?
3. If the potassium permanganate is mixed with an alkene, what happens in the test tube?

Experiment 5: Extraction

Experiment Procedure

Creating the Lemon Peel Extract: Peel one lemon and discard the fruit's core. Tear the lemon peel into small pieces and place them into an empty jar. Using a 50-mL test tube, measure out 50 mL of 91% isopropanol and add it to the jar of lemon peels. Put the lid on the jar and shake it vigorously for 15-20 minutes. After shaking, let the lemon peel solution settle for 5 minutes. While you're waiting, fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of another empty jar. Filter the lemon peel solution using the filtration setup that you just constructed (see picture below). The resulting liquid (filtrate) shouldn't have any solid pieces floating in it. Discard the used lemon peels. Set the lemon filtrate aside. **Take a picture of the lemon filtrate.**



Creating the Cloves Extract: Add two tablespoons of ground cloves to an empty jar. Using a 50-mL test tube, measure out 50 mL of 91% isopropanol and add it to the jar of ground cloves. Put the lid on the jar and shake it vigorously for 15-20 minutes. After shaking, let the clove solution settle for 5 minutes. Create a filtration setup like the one you made for the lemon peels. Fold two coffee filters in half (twice) then place them inside a plastic funnel. Set the plastic funnel on top of another empty jar. Filter the clove solution using the

filtration setup. The resulting filtrate shouldn't have any solid pieces floating in it. Discard the used clove powder. **Take a picture of the clove filtrate.**

Evaporation & Testing: Place a paper towel over both jars containing your filtrates (lemon peel & cloves). You can secure the paper towel with a rubber band. The paper towel should fully cover the mouth of the jars to keep out solid debris. Let both jars sit in a window sill for 24 hours. Over the 24 hour period, some of the isopropanol will evaporate and your extracts will become more concentrated.
After the 24 hour period, obtain two 15-mL test tubes. Using two different plastic pipets, place 2 mL of lemon peel extract in one test tube and 2 mL of clove extract in the other test tube. Add one drop of $KMnO_4$ solution to each test tube and swirl. Record what you observe happening in the test tubes after the $KMnO_4$ solution was added. Let the mixture sit for 5-10 minutes. **Take a picture of the test tubes after you let them sit for 5-10 minutes.**

Waste Disposal: Test tubes, funnels and jars should be washed thoroughly with soap and water, dried then placed back in the chemical/equipment kit. The liquid extract can be washed down the sink with copious amounts of water. The solids and filter paper can be thrown away in the trash can.

Experiment 5: Extraction

Photo Logbook **Name:**

1. Insert a picture of the lemon peel filtrate.

2. Insert a picture of the clove filtrate.

3. Insert a picture of the test tubes after one drop of $KMnO_4$ solution was added and left to sit for 5-10 minutes.

Experiment 5: Extraction

Post-Lab Questions **Name:**

1. What did you observe happening after one drop of $KMnO_4$ solution was added to your extracts? What does this tell you about the extracts?

2. One of the main organic components in lemon peel is limonene. Use Chemsketch to draw the structure of limonene. Does limonene contain an alkene functional group?

3. One of the main organic components in cloves is eugenol. Use Chemsketch to draw the structure of eugenol. Does eugenol contain an alkene functional group?

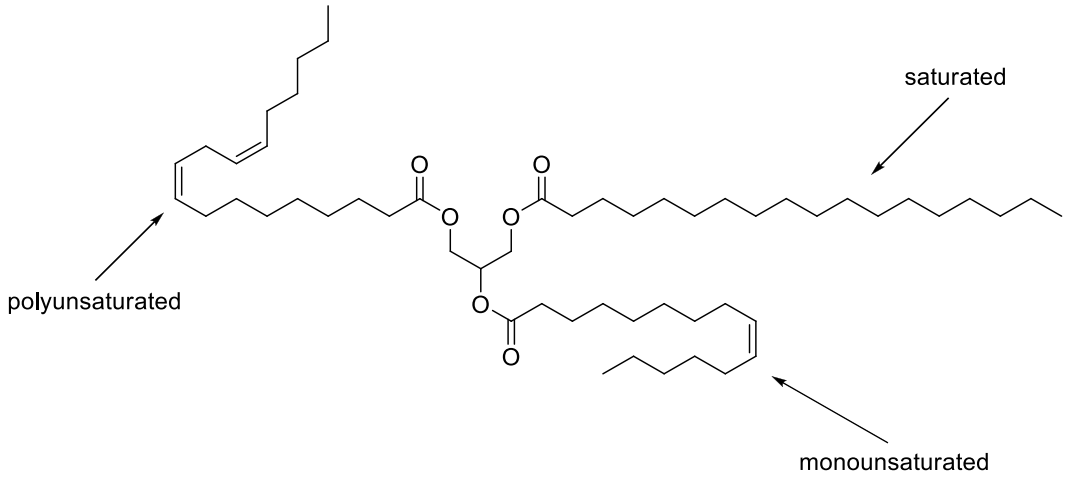
4. Over time many extracts, essential oils, and fragrances will lose their smell. Why?

5. Hexane would be a useful solvent for extracting limonene from lemon peels but not eugenol from cloves. Explain.

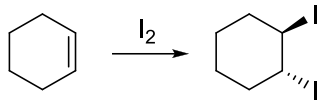
6. Organic solvents can be broken down into categories like polar and non-polar. They can also be categorized as protic and aprotic. What does protic and aprotic mean? Give an example of a protic and aprotic solvent.

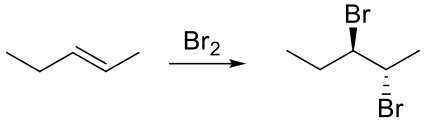
Experiment 6: Saturated & Unsaturated Oils

Oils (or triacylglycerides) contain three ester functional groups that connect one glycerol molecule to three fatty acids. The fatty acids can be saturated or unsaturated. Saturated fatty acids contain only carbon-carbon single bonds in their hydrocarbon chain. Unsaturated fatty acids contain one or more carbon-carbon double bonds (alkenes) in their hydrocarbon chain. Depending on the oil, the saturated and unsaturated fat contents can vary. For example, olive oil contains ~90% unsaturated fat while coconut oil only contains ~10%. Unsaturated fats can be broken down into two categories: monounsaturated and polyunsaturated. Monounsaturated fatty acids contain only one alkene while polyunsaturated fats contain two or more alkenes. Shown below is an example of an oil. This oil was made from three different types of fatty acids.



Alkenes can undergo reactions with halogens. In this experiment, you are given three different oils. The oils will contain only saturated fats or varying amounts of unsaturated fats. Unsaturated fats contain alkenes which will react with iodine by adding across the double bond. If this occurs, the characteristic purple-brown color of iodine will disappear from the solution. You will need to monitor the three different oils and look for the disappearance of the iodine color.





When Br_2 or I_2 adds across an alkene, the reaction occurs with *anti stereochemistry.* The two bromine (or iodine) atoms add on opposite faces of the double bond (notice the dashes and wedges in the products).

Materials
Three 15-mL test tubes

Chemicals
Oil A (in vial)
Oil B (in vial)
Oil C (in vial)
Iodine tincture solution (in dropper bottle)

Safety: Make sure to wear your safety goggles when handling any chemicals. The iodine tincture solution will stain both clothes and skin.

Experiment 6: Saturated & Unsaturated Oils

Pre-lab Exercise **Name:**

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Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. You must wear your ( ) when handling any chemicals.
    b. Iodine tincture can ( ).
2. If an oil is unsaturated, what will happen when you add the iodine tincture?
3. When Bromine or Iodine adds across an alkene double bond, what is the stereochemistry?

Experiment 6: Saturated & Unsaturated Oils

Experiment Procedure

Unsaturation Test: Label three 15-mL test tubes with the letters: A, B, & C. To test tube A, pour in ~2.5 mL of Oil A. To test tube B, pour in ~2.5 mL of Oil B. To test tube C, pour in ~2.5 mL of Oil C. To each test tube, add three drops of the iodine tincture solution. **DO NOT SHAKE THE TEST TUBES.** The iodine solution should sink to the bottom of the tubes. Observe all three test tubes over a one hour period. **Take pictures of all three test tubes after 10 minutes, 20 minutes, 30 minutes and 1 hour.** After 1 hour, you can dispose of the oil solutions.

Waste Disposal: The test tubes should be washed thoroughly with soap and water, dried then placed back in the chemical/equipment kit. The oil waste can be washed down the sink with copious amounts of water.

Experiment 6: Saturated & Unsaturated Oils

Photo Logbook **Name:**

1. Insert a picture of the three test tubes 10 minutes after the iodine was added.

2. Insert a picture of the three test tubes 20 minutes after the iodine was added.

3. Insert a picture of the three test tubes 30 minutes after the iodine was added.

4. Insert a picture of the three test tubes 1 hour after the iodine was added.

Experiment 6: Saturated & Unsaturated Oils

Post-Lab Questions **Name:**

1. Which of the oils contain unsaturated fats (which of the oils went colorless)?

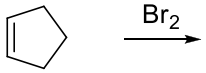
2. Which of the oils contain only saturated fats (which of the oils had no color change)?

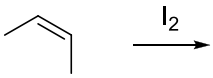
3. Why did the oils containing only saturated fats not react with the iodine solution?

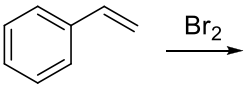
4. Using Chemsketch, draw glycerol.

5. Using Chemsketch, draw lauric acid, linoleic acid, and oleic acid. Label each as a saturated, monounsaturated, or polyunsaturated fatty acid.

6. Using Chemsketch, draw the products of the following reactions. Include stereochemistry when necessary.

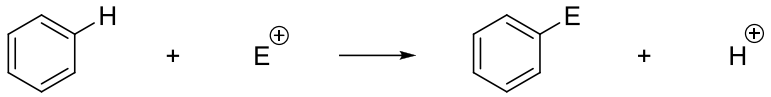
A) 

B) 

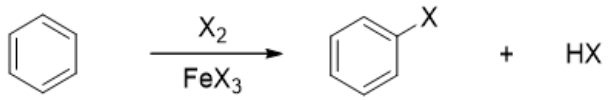
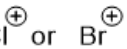
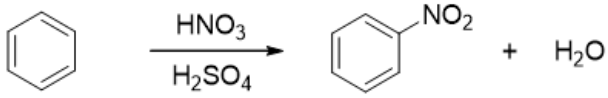
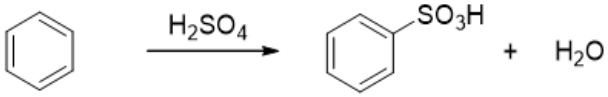
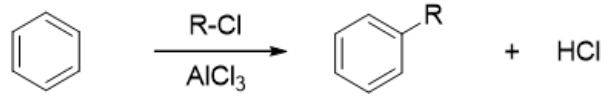
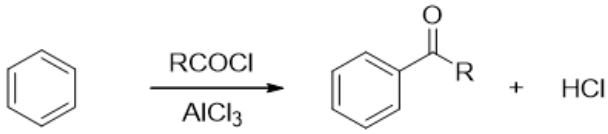
C) 

Experiment 7: Electrophilic Aromatic Substitution

Electrophilic Aromatic Substitution (EAS) is one of the most important reactions as it allows for placement of many different functional groups on an aromatic ring. The overall reaction is shown below, where some compound (labelled as "E" for "electrophile") is added on to a benzene or aromatic ring.

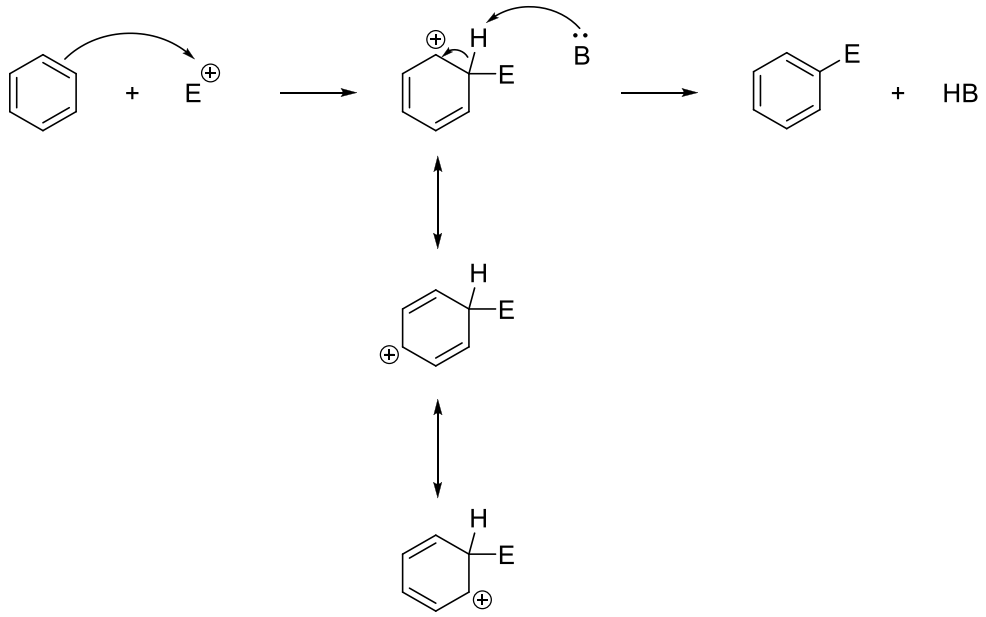


There are many different groups that can be added on as the electrophile and they each have specific reaction conditions to create the electrophile part. Examples of electrophile groups that can be added on are shown in the chart below:

Reaction Type	Typical Equation	Electrophile (E^+)
Halogenation		
Nitration		
Sulfonation		
Friedel-Crafts Alkylation		
Friedel-Crafts Acylation		

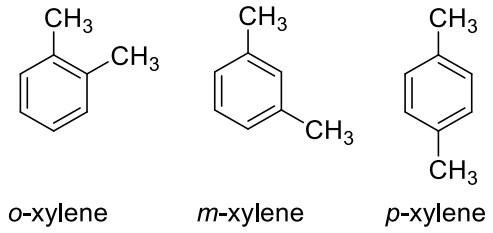
One of the really interesting aspects to Electrophilic Aromatic Substitution occurs when you add **more than one substituent** on to the aromatic ring, because we find that the first group added directs **where** a second group gets added.

Reaction mechanism for how EAS works: When an electrophile is added on to the aromatic ring, the reaction forms an intermediate as shown below. The intermediate can be stabilized by different resonance structures. The intermediate complex loses an H^+ as the last step to regain the aromatic ring.



Defining the *ortho-, meta-* and *para-* sites on an aromatic ring: When two substituents are placed on a benzene ring, we can define the relative spacing of the substituents using the terms *ortho-, meta-* and *para-*.

- *Ortho-*, (abbreviated as *o-*) means the two substituents are at the 1,2 positions around the ring.
- *Meta-*, (abbreviated as *m-*) means the two substituents are at the 1,3 positions around the ring.
- *Para-*, (abbreviated as *p-*) means the two substituents are at the 1,4 positions around the ring.

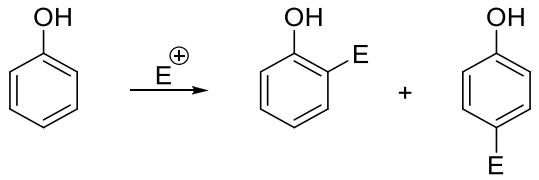


Directing effects of a substituent: When we add more than one substituent on to a benzene ring, we find that the first group added determines where a second group gets added. We also find patterns on whether the first group activates the aromatic ring and makes it more likely to react, or deactivates the ring, making it less likely to react.

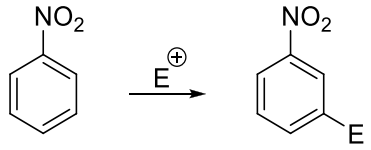
The patterns we observe are shown below:

Activating Substituents Ortho/Para Directors	Deactivating Substituents Meta Directors	Deactivating Substituents Ortho/Para Directors
$-O^{\ominus}$	$-\overset{\oplus}{S}R_2$	
$-NH_2$	$-COOH$	
$-OH$	$-NO_2$	$-F$
$-NR_2$	$-COOR$	
$-OR$	$-SO_3H$	$-Cl$
$-NHCOR$	$-CONHR$	
$-OPh$	$-SO_2R$	$-Br$
$-R$	$-COR$	
$-OCOR$	$-CHO$	$-I$
$-Ph$	$-CN$	
	$-\overset{\oplus}{N}R_3$	
	$-\overset{\oplus}{P}R_3$	

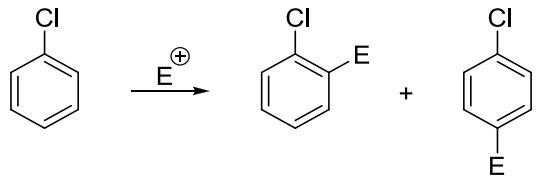
For example, an –OH group on the benzene ring will activate the ring (make it more likely to react) and direct the next group to either the *ortho*- position or the *para*- position.



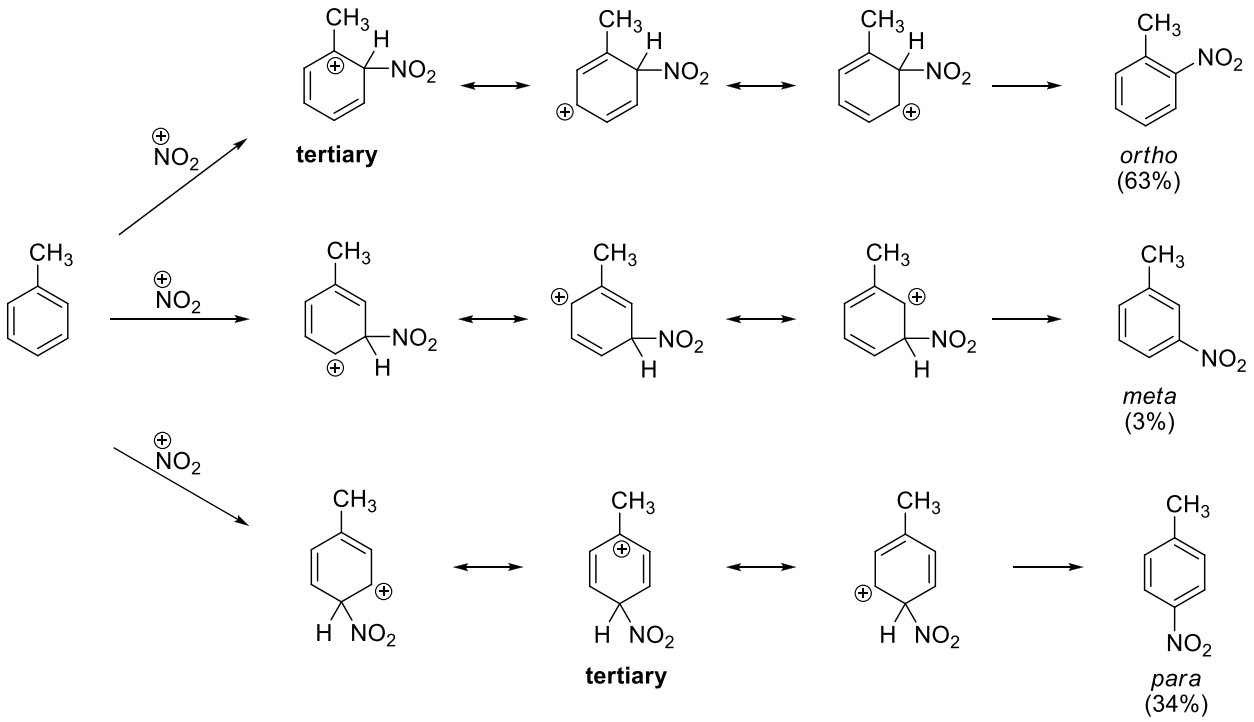
An $-NO_2$ group on the benzene ring will deactivate the ring (make it less likely to react) and direct the next electrophile group to the *meta*- position.



And then a halogen like chlorine, will deactivate the ring but direct *ortho*- and *para*- for the next substituent.



Why do we see these directing effects: We see these directing effects because of the intermediate formed in the reaction and its resonance structures. Some of the resonance structures are more stable than others so it becomes easier to add the second substituent at a particular location. Below are the resonance structures shown for toluene when a second group is added. The *ortho*- and *para*- positions produce a more stable resonance structure because the methyl group can donate electrons and stabilize the positive carbocation when it is on the carbon next door. This more stable resonance structure causes a second reaction to happen at the *ortho*- and *para*- locations. Each of the EAS substituents can have these resonance structures that show why they direct reaction to a particular location on the ring.


CH3
NO2
H
tertiary
ortho
(63%)
meta
(3%)
para
(34%)

Experiment 7: Electrophilic Aromatic Substitution

Pre-lab Exercise **Name:**

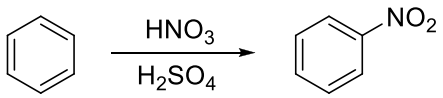
This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Fill in the blanks between parentheses to answer the following questions:
   a. ( ) means the two substituents are at the 1,3 positions around the ring.
   b. An $-NO_2$ group on the benzene ring will ( ) the ring (make it less likely to react)…
2. Which of the following groups is a deactivating substituent and prefers an ortho/para orientation? Underline or circle your answer.

   A. –OH B. $-NH_2$ C. $-CO_2H$ D. –F

3. What type of reaction (reaction name) is shown below? Hint: the electrophile is NO_2^+.



Experiment 7: Electrophilic Aromatic Substitution

Experiment Procedure

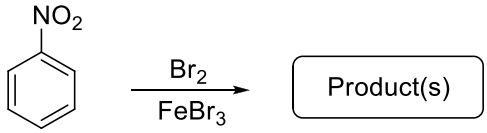
Predict the directing effects of substituents on the benzene ring.

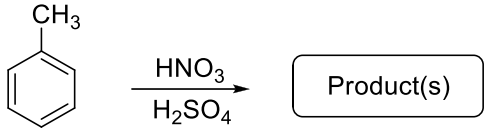
For each compound shown below, do the following:

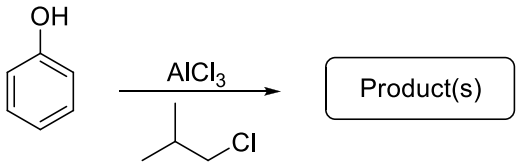
a. Predict if the substituent would activate or deactivate the ring for the next reaction.

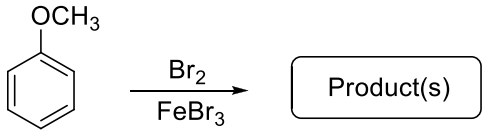
b. Predict where the next electrophile is most likely to add (*ortho*-, *para*- as a group or *meta*-).

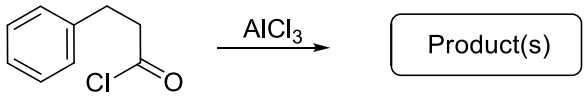
c. Draw the predicted product(s) of the reactions in ChemSketch. Copy and paste the 3-D image into your logbook for grading.

(i) 

(ii) 

(iii) 

(iv) 

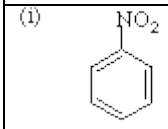
(v) 

Experiment 7: Electrophilic Aromatic Substitution

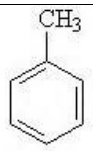
Photo Logbook **Name:**

For each reaction shown below, fill in the answers. Copy and paste your 3D image from Chemsketch for the predicted products (do both *ortho*- and *para*- if it is appropriate).

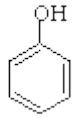
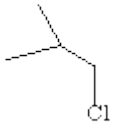
Remember that you are looking at the group ALREADY ATTACHED to the benzene ring to determine the activation and directing effects. The next electrophile being added will determine what the product looks like.

Initial Compound	Electrophile being added	Activate or Deactivate?	Directing effects (predict o-,p- or m-)
(i) 	Br^+		

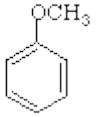
Copy your image(s) for predicted products here:

Initial Compound	Electrophile being added	Activate or Deactivate?	Directing effects (predict o-,p- or m-)
 ii)	NO_2^+		

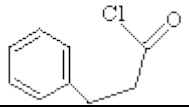
Copy your image(s) for predicted products here:

Initial Compound	Electrophile being added	Activate or Deactivate?	Directing effects (predict o-,p- or m-)
(iii) 			

Copy your image(s) for predicted products here:

Initial Compound	Electrophile being added	Activate or Deactivate?	Directing effects (predict o-,p- or m-)
(iv) 	Br^+		

Copy your image(s) for predicted products here:

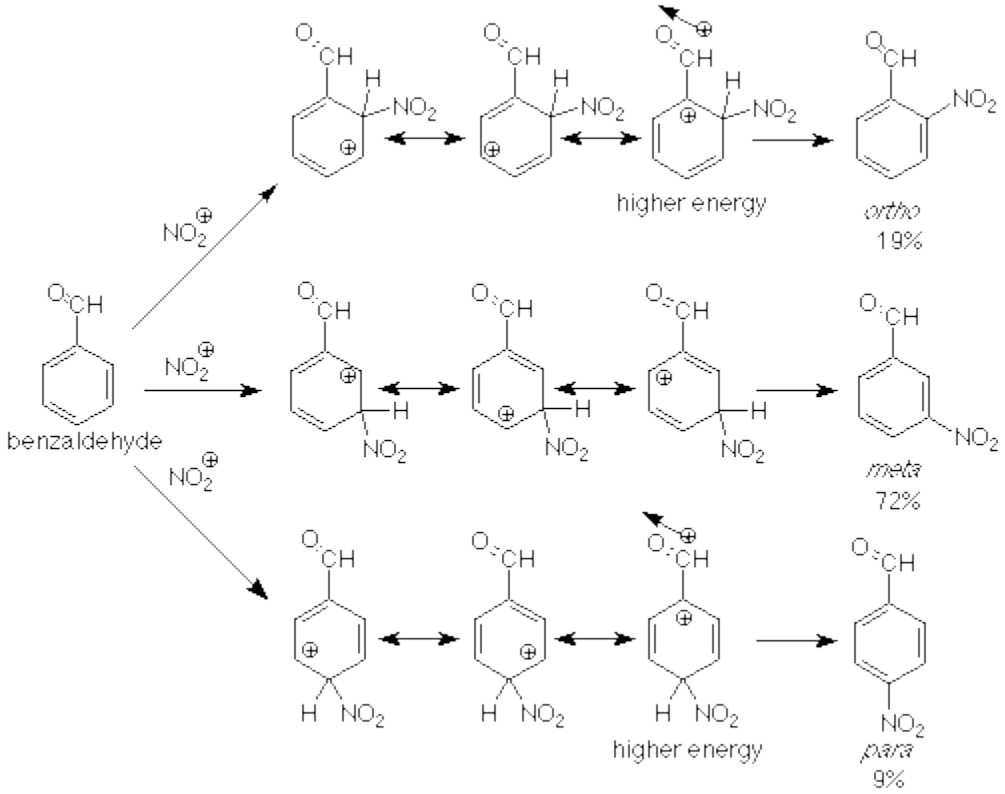
Initial Compound	Electrophile being added	Activate or Deactivate?	Directing effects (predict o-,p- or m-)
(v) 	Side chain with Cl on it		

Copy your image(s) for predicted products here:

Experiment 7: Electrophilic Aromatic Substitution

Post-lab Questions **Name:**

1. We had discussed in the introduction that ortho-,para directing groups have more stable resonance structures for the intermediate. Shown below are the resonance structures for addition to benzaldehyde. Explain why the ortho- and para- choices lead to higher energy resonance structures.



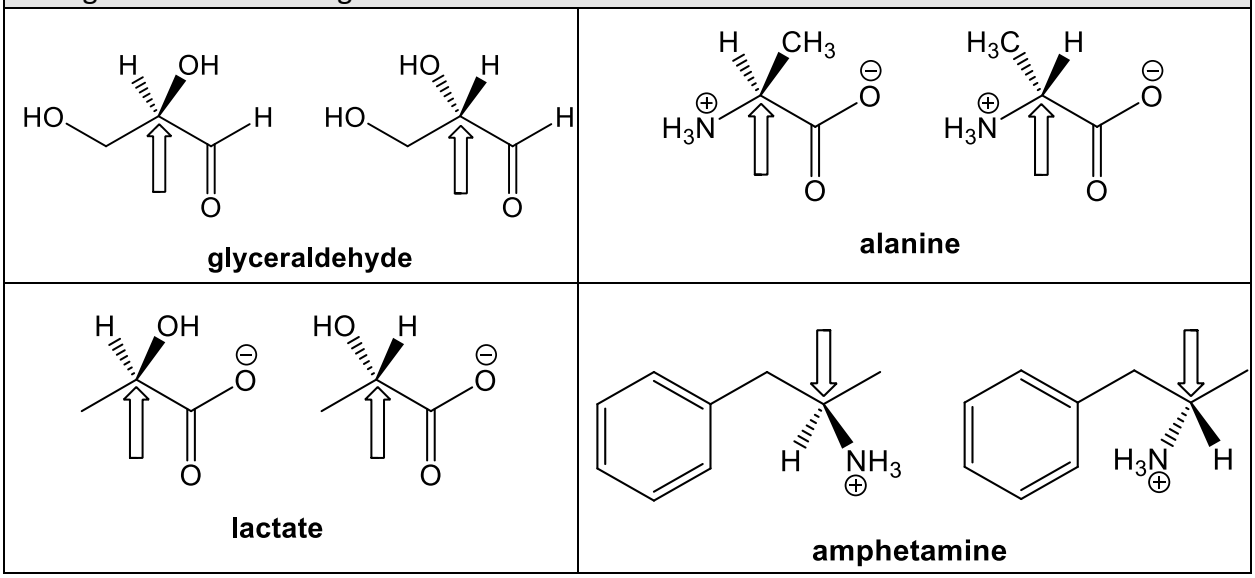
Experiment 8: Stereochemistry and Identification of R and S

Note: Before attempting this lab, you must review the Chemsketch Walkthrough instructions provided! You will have difficulty if you do not first educate yourself on how ChemSketch works.

Chemical molecules can have a variety of different isomers. The purpose of this modeling lab is to focus on stereoisomers, or different configurations of atoms arranged around a chiral carbon. There are three steps to designating a stereoisomer configuration.
Step 1: Identifying a chiral carbon.
Step 2: Ranking the four groups bonded to the chiral carbon.
Step 3: Designating the stereoisomer as R or S configuration based on how the groups are bonded.

Step 1–Identifying a chiral carbon: A carbon is defined as chiral if there are 4 distinct and different groups bonded to it. In the case of a ring structure, a carbon can still be defined as chiral if the portions of the ring are different on one side as opposed to another. Examples of chiral and non-chiral carbons are shown below in Figure 1.

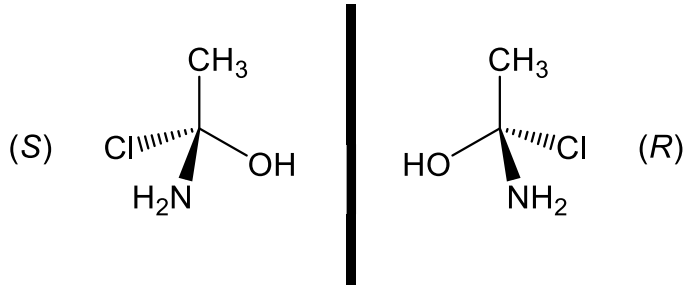
Figure 1. The arrow indicates a chiral carbon in each compound. Four different groups are bonded to the chiral carbon. Other carbons shown in these structures are not chiral, because they do not have 4 different groups bonded. Note that each example shows two different configurations of bonding around the chiral carbon.



Step 2–Ranking the four groups bonded to the chiral carbon: If a carbon is designated as chiral and has four different groups bonded to it, then we can rank each group according to the Cahn-Ingold-Prelog (CIP) system of priority. The rules for priority ranking go as follows:

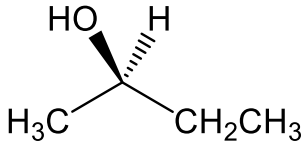
1. Look at the atom directly bonded to the chiral carbon. **Whichever atom has the highest atomic number (Z value) is ranked highest, down to the lowest atomic number ranked 4th.** For example, chlorine in the example below has the highest atomic number and would be ranked #1. Carbon has the lowest atomic number and would be ranked #4 (with O ranked #2 and N ranked #3).

   Example:



2. **If two groups tie with their atomic number, then we continue to the next set of atoms to see if there is a difference in priority.** With the example below, the oxygen ranks #1; the hydrogen ranks #4 around a chiral carbon. The two carbons bonded have identical atomic number so we move to the next group of atoms bonded. The methyl group ($-CH_3$) is bonded to 3 hydrogens; the ethyl group ($-CH_2CH_3$) is bonded to 2 hydrogens and 1 carbon. The ethyl group would therefore rank higher (because the 1C/2H combo has a higher overall atomic number than the 3H combo).

   Example:



3. **If a group contains double or triple bonds, then we count the double/triple bonded atom twice (or three times for a triple bond).** We then use the same atomic number ranking to assign priorities. In the structure below, N ranks as #1; H ranks as #4 around a chiral carbon. The other two groups are both carbon directly bonded so we go one step further out. The group heading up has a carbon with 1 oxygen and 2 hydrogens bonded to it; the group to the right has 1 oxygen single bonded and 1 oxygen double bonded (so we count it twice). This means the group to the right has a higher priority and is designated #2; the group heading up is ranked #3 in priority.

   Example:

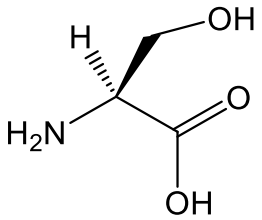
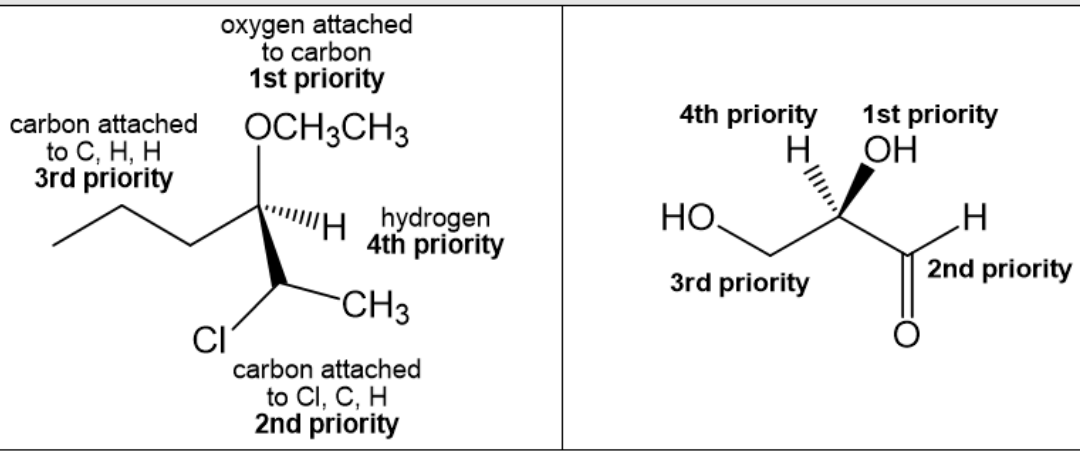
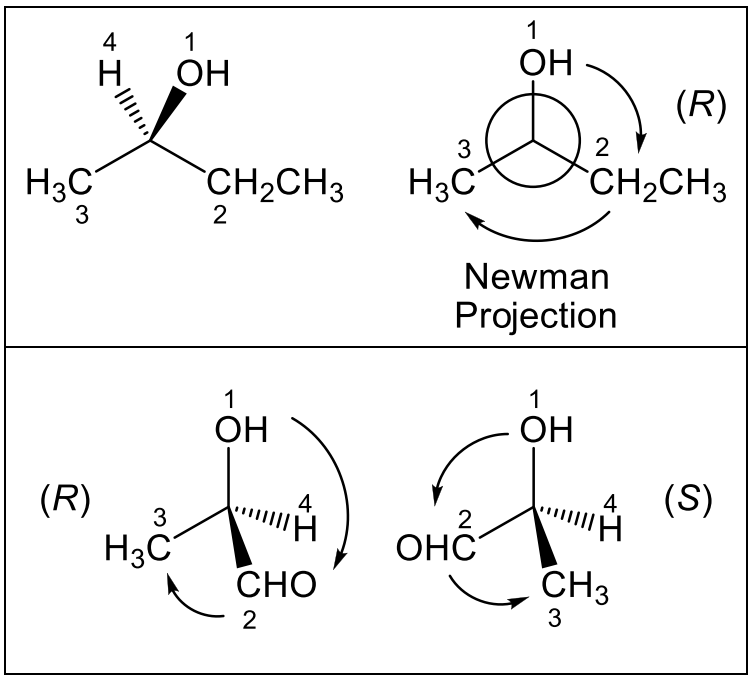


Figure 2. Examples of priority ranking when groups have multiple bonds and other atoms attached.



Step 3–Designating the stereoisomer as R or S configuration based on how the groups are bonded: A chiral carbon is then designated as **R** ("rectus"; Latin for "right") or **S** ("sinister"; Latin for "left") based on whether the groups are attached in a clockwise orientation (R) or a counter-clockwise orientation (S). To determine this, orient the molecule so that the #4 designated group is hiding in the back away from you. If the other 3 groups are arranged in a clockwise configuration, then the isomer is designated "R"; if the other 3 groups are arranged counter-clockwise, then the isomer is designated "S". Examples are shown below:



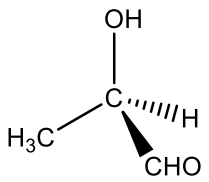
Experiment 8: Stereochemistry and Identification of R and S

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Fill in the blanks between parentheses to answer the following questions:
    a. Before attempting this lab, you must review the ( ) instructions provided!
    b. A carbon is defined as ( ) if there are 4 distinct and different groups bonded to it.
2. On the compound shown below, what group would have first priority? Underline or circle your answer.



A. –OH B. $-CH_3$ C. –CHO

D. –H

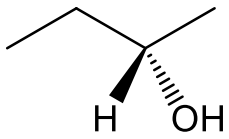
3. We rank groups on each chiral carbon according to what system?

Experiment 8: Stereochemistry and Identification of R and S

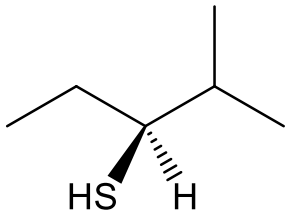
Experiment Procedure

Part A–Identify the R or S designation around chiral carbons: For each of the following compounds, label in order of priority the four groups around a chiral carbon (with #1 being the highest and #4 being the lowest priority). **Note: some compounds may have more than one chiral carbon shown**. Designate the stereoisomer as R or S based on this ranking. Note: you will have to orient the molecule with the #4 group hiding in the back to do this. You may find it helpful to re-draw the compound with #4 in the back so you can designate R or S.

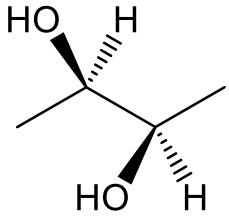
(a)



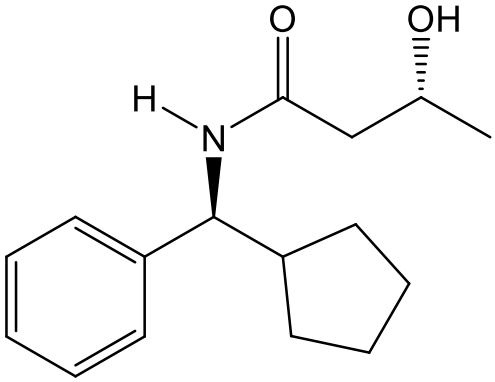
(b)



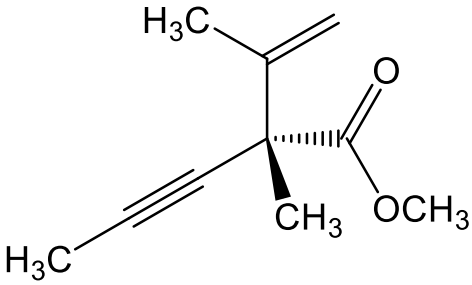
(c)



(d)

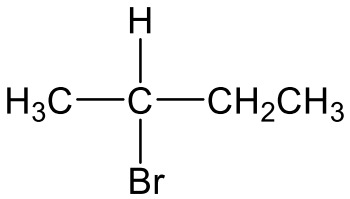


(e)

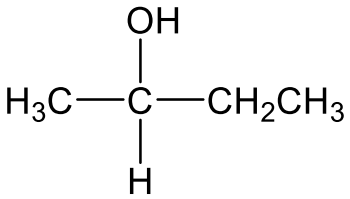


Part B–Drawing stereoisomers: Draw both the R and the S stereoisomers in ChemSketch for the following compounds. For full credit, clearly show the stereochemistry around each chiral carbon(s) and label the isomers as R and S. Create the 3D image for upload in your photo log.

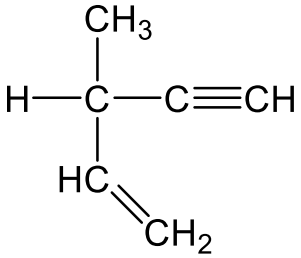
(a)



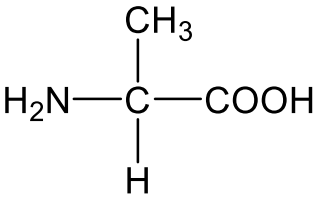
(b)



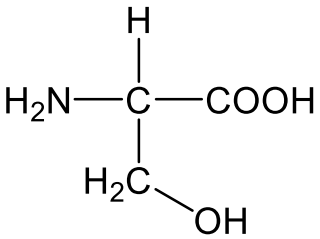
(c)



(d)



(e)

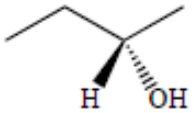
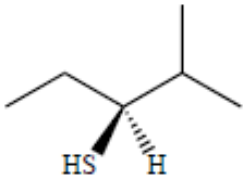
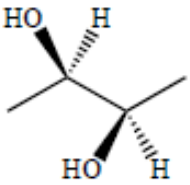
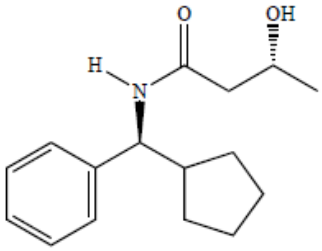
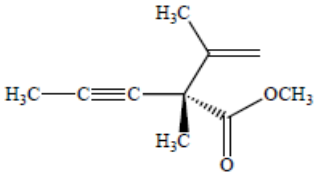


Experiment 8: Stereochemistry and Identification of R and S

Photo Logbook/Post-lab Questions **Name:**

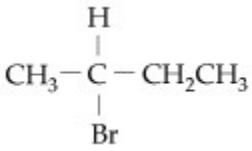
Part A: Identify the R or S designation around chiral carbons

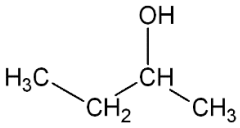
1. For each of the following compounds, label in order of priority the four groups around a chiral carbon (with #1 being the highest and #4 being the lowest priority)(**Note: some compounds may have more than one chiral carbon shown**). Designate the stereoisomer as R or S based on this ranking. (Note: you will have to orient the molecule with the #4 group hiding in the back to do this. You may find it helpful to re-draw the compound with #4 in the back so you can designate R or S).

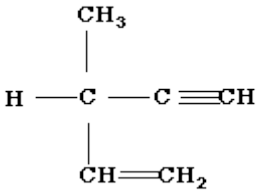
Compounds	Priority group label	Stereoisomer Designation
(a) 	What is the ranking number for Ethyl in this compound?	
(b) 	What is the ranking number for ethyl in this compound?	
(c) 	What is the ranking number for OH for the first chiral carbon (the one on the left) and the second chiral carbon (the one on the right)	
(d) 	What is the ranking number for the benzene ring (the one with double bonds)?	
(e) 	What is the ranking number for the triple bonded group?	

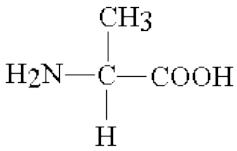
Part B: Drawing stereoisomers.

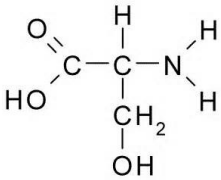
1. Draw both the R and the S stereoisomers in ChemSketch for the following compounds. For full credit, clearly show the stereochemistry around each chiral carbon(s) and label the isomers as R and S. **Insert the 3D image for each stereoisomer below the compound!**

a. 

b. 

c. 

d. 

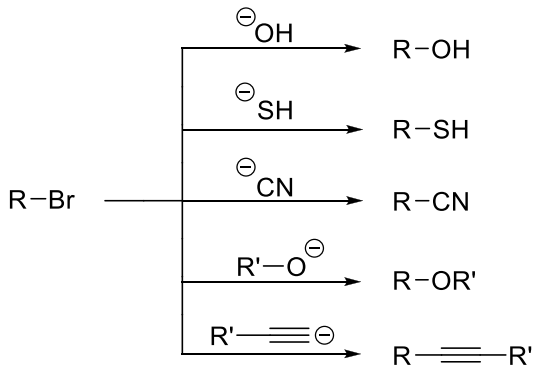
e. 

Experiment 9: Alkyl Halides and S_N1/S_N2 Reactions

Stereochemistry: Chiral carbons can be designated as R or S stereochemistry. Review section 6.6 of your textbook for review on designating R or S.

S_N1 and S_N2 reactions of alkyl halides: Substitution reactions are an important class of reactions because of their synthetic utility and importance in revealing the mechanism of certain organic reactions. Substitution reactions allow the introduction of a variety of functional groups into organic molecules.

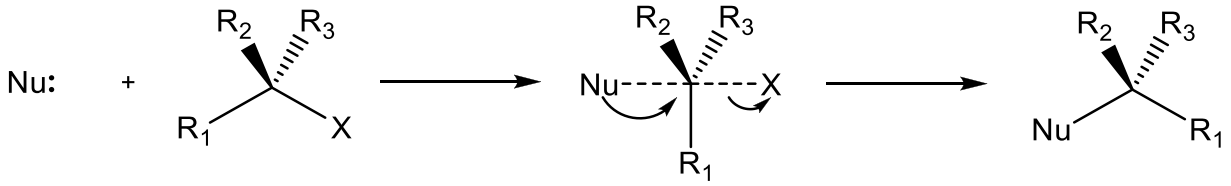
Figure 1: Substitution reactions can form many possible products.



Chemists have studied the mechanism of substitution reactions in great detail. The results of these studies have indicated that substitution reactions can be broadly divided into two mechanistic types, called S_N2 and S_N1.

S_N2 (Substitution, Nucleophilic, 2nd order) reactions proceed via a one-step mechanism in which the incoming nucleophile attacks the electrophilic carbon center from the side opposite the leaving group. This reaction mechanism results in inverted stereochemistry around the chiral center. So if you start with an "R" alkyl halide, the product will be "S" or vice versa. Since the reaction happens as a one-step mechanism, substrates that have only hydrogen or one R group attached to the alkyl halide will react best.

Figure 2: S_N2 reaction mechanism



Factors that determine when S_N2 occurs:

1. **Alkyl halide group size:** Because this reaction has to have a nucleophile attack from behind and it goes through a 5-membered intermediate, small alkyl halides react best. The order of reactivity for S_N2 reactions is:

   Methyl > 1° > 2° > 3°

2. **Strength of nucleophile:** Stronger nucleophiles are ones that have a negative charge associated with them. Stronger nucleophiles react according to S_N2 best since they initiate the reaction to occur by attacking the back of the alkyl halide. Weak nucleophiles (neutral compounds with no negative charge) do not react well via S_N2.

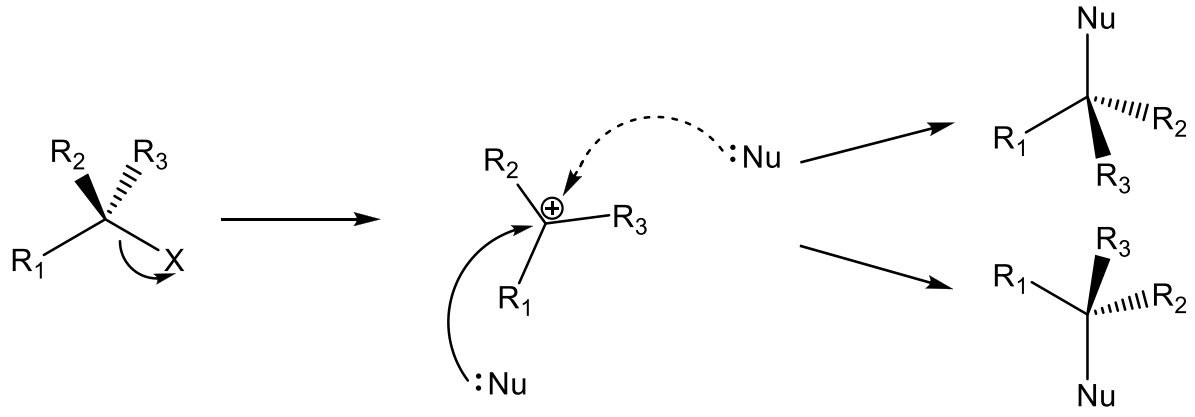
S_N1 reactions (Substitution, Nucleophilic, 1st order) proceed via two steps:

(1) Slow dissociation of the C-X bond to form an intermediate carbocation and

(2) A fast second step in which the C-Nu bond is formed.

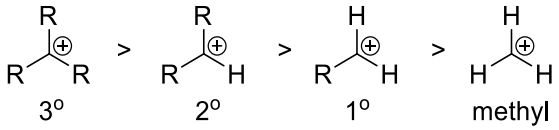
Since the intermediate carbocation is trigonal planar, the nucleophile can attack with equal probability from either side. This will result in equal amounts of each enantiomer being produced as products. This means if you start with an "R" alkyl halide, you will get half "R" product and half "S" product (in theory).

Figure 3: S_N1 reaction mechanism



Factors that determine when S_N1 occurs:

1. **Alkyl halide group size**: The S_N1 reaction involves a carbocation as the intermediate. Markovnikov's rule states that carbocation stability runs:



S_N1 reactions will tend to occur best with tertiary alkyl halides or other stable carbocations.

2. **Strength of nucleophile:** Since the carbocation is formed independently as a first step, a weak nucleophile works well for S_N1. Weak nucleophiles are ones that do not have a negative charge and are neutral compounds.

Experiment 9: Alkyl Halides and S_N1/S_N2 reactions

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. List what is important about alkyl halide compound size and nucleophile to make S_N2 occur.
2. List what is important about alkyl halide compound size and nucleophile to make S_N1 occur.
3. How can you decide if a nucleophile is strong or weak?

Experiment 9: Alkyl Halides and S_N1/S_N2 reactions

Experiment Procedure

ChemSketch: Open up ChemSketch. For ***each of the reactions listed below***, do the following:

A. Draw the *reactant*, including listed stereochemistry, select model and under Tools, hit 3-D Optimization. When running 3-D Optimization, make sure it counts the Hydrogens. Convert the reactant image to 3-D, orient the image so that the least ranked substituent is in the back and copy the image into your photo log

B. For each reaction, draw the organic *product(s)* for each reaction including predicted stereochemistry, select model and under Tools, hit 3-D Optimization. Convert the product image to 3-D, orient the image so that the least ranked substituent is in the back and copy the image into your photo log

C. ***Identify reaction type*** (S_N1 or S_N2) and ***product stereochemistry*** on your worksheet.

Reaction #	
1	(*R*)-2-butylchloride + CH_3S^- $\rightarrow$
2	(*R*)-3-chloro-3-methylhexane + H_2O $\rightarrow$
3	(*S*)-2-bromohexane + CH_3COO^- (sodium acetate) $\rightarrow$
4	(*R*)-2-iodopentane + ^-OH $\rightarrow$
5	methyl bromide + ^-CN $\rightarrow$
6	(*S*)-2-bromobutane + NH_3 $\rightarrow$
7	(*R*)-2-bromooctane + $CH_3CO_2^-$ $\rightarrow$
8	(*R*)-2-bromobutane + CH_3OH $\rightarrow$

Experiment 9: Alkyl Halides and S_N1/S_N2 reactions

Photo Logbook **Name:**

Reaction #	
1	R-2-butylchloride + CH_3S^- →
2	R-3-chloro-3-methylhexane + H_2O →
3	S-2-bromohexane + CH_3COO^- (sodium acetate) →
4	R-2-iodopentane+ OH^- →
5	methyl bromide + CN^- →
6	S-2-bromobutane + NH_3→
7	R-2-bromooctane + $CH_3CO_2^-$ →
8	R-2-bromobutane + CH_3OH →

Experiment 9: Alkyl Halides and S_N1/S_N2 Reactions

Post-Lab Questions **Name:**

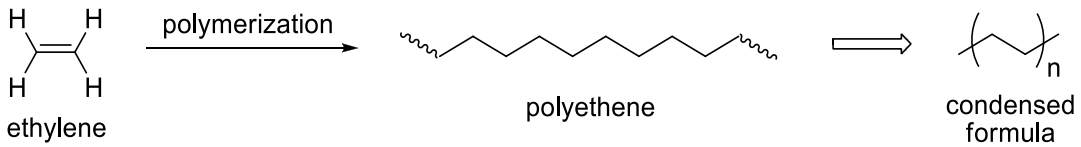
1. Complete the following table. For each reaction, classify the type of reaction (S_N1 or S_N2) and identify the stereochemistry of the product(s).

Reaction #	Reaction type	Stereochemistry of Product
1		
2		
3		
4		
5		
6		
7		
8		

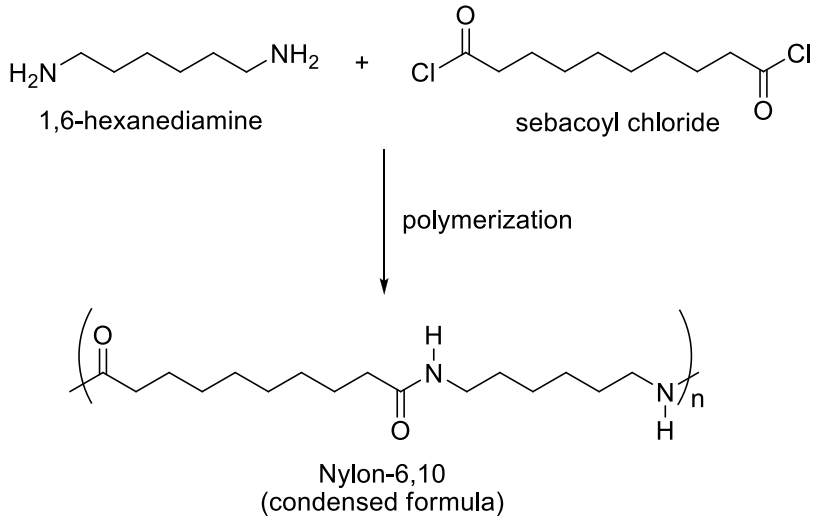
Experiment 10: Making Polymers

In this experiment you will be making several polymers. The term polymer is from the Greek terms: *polys* – many, and *meros* – part. A linear polymer is a single continuous chain formed by one monomer molecule combining with other monomer molecules in a sequence. This results in a very long molecule which may be made up of 1,000–10,000 monomer molecules having a molecular weight on the order of 100,000– 00,000 g/mol or more. The polymer chains are not chemically bonded to another polymer chain and are relatively free to move.

There are two main approaches to polymerization. **Chain growth** is shown below and uses a double bond to bind monomers together. Depending on the reactants, the mechanism can occur via a cation intermediate, an anion intermediate or a free radical intermediate.



Step growth or condensation polymerization involves two different reactants and releases a small molecule, typically water in the reaction. An example of step growth polymerization is shown with the Nylon reaction below. Nylon-6,10 is a linear polymer that is formed through a condensation reaction from the monomers 1,6-hexanediamine and sebacoyl chloride. This nylon is designated as “6,10” because it is made from a diamine with six carbon atoms and a dicarboxylic acid derivative (sebacoyl chloride) which contains ten carbon atoms. Nylon is extremely strong and is used for climbing rope and parachutes.



A cross-linked polymer is made from at least one monomer which has a third site at which a chemical bond may form. This results in the polymer chains being chemically bonded to each other and they cannot move independently of each other. Such polymers are usually much harder or more viscous than a similar linear polymer. The molecular weight of a cross-linked polymer can be huge, because a cross-linked polymer is one gigantic polymer molecule. For example, a tire is made from a cross-linked polymer and could be considered to be one single enormous molecule.

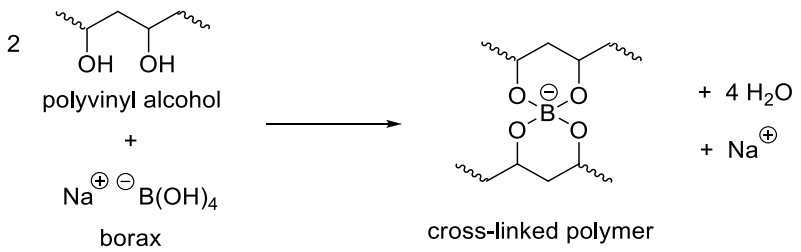
The physical properties of polymers help determine the applications and use of that plastic. The typical tests for polymers to use to compare one polymer to another are: tensile strength, glass transition temperature (T_g), elongation, and tear resistance. Precise equipment is used to measure properties such as tensile strength and glass transition temperature, but these properties can also be approximated with simple equipment.

Crystalline polymers have individual chains that are folded and packed regularly in an ordered fashion. The polymer chains are usually long; therefore the ordered arrangement is not perfect. Examples of crystalline polymers are polyethylene and polypropylene. Polypropylene is used in yarn for carpet, knitted fabrics and liners for disposable diapers. Polyethylene is used in packaging materials, in wire and cable insulation and milk cartons. Low density polyethylene (LDPE) is used for plastic sandwich bags.

Amorphous polymers have unordered regions. The polymer chains are arranged randomly and can be entangled. Polystyrene and polycarbonate are examples of amorphous polymers. Polystyrene is used in fast food packaging, egg containers, and mirror and picture frames. Polycarbonate is used in automobile taillight lenses, bumpers, and drapery fixtures.

Some polymers have halogens as part of their structure. Polymerization of vinyl chloride (chloroethene) produces a polymer similar to polyethylene, but having chlorine atoms at alternate carbon atoms on the chain. Polyvinyl chloride (PVC) is rigid and somewhat brittle. About two-thirds of the PVC produced annually is used in the manufacture of pipe. Another example is polytetrafluoroethylene which is used for cookware as Teflon.

In this chemical procedure, a polymer called polyvinyl alcohol is used. In this polymer, vinyl alcohol has already been polymerized from many alcohol monomers by chain growth polymerization. A second chemical, borax (sodium borate) is added to the polyvinyl alcohol to crosslink the polymer chains. Crosslinking limits the movement of the polymer and does not allow the polyvinyl alcohol polymers to move freely or independently of each other. Cornstarch is also a long polymer chain. The addition of cornstarch to the mixture gives it more solidity. The solutions that you will make are a 55 % Elmer's glue solution (which contains polyvinyl alcohol) and a 4% borax solution. You will construct two different polymer balls that have different physical properties.



Materials

Plastic spatula
Jar
Measuring cup
A teaspoon
A tablespoon
Three small Ziploc bags

Chemicals

Elmer's Glue (in test tube)
Borax (in a Ziploc bag)
Cornstarch
Food coloring (optional)

Safety: Make sure to wear your safety goggles when handling any chemicals. The food coloring may stain your hands or clothes so you may want to wear gloves and an apron when doing this experiment.

Experiment 10: Making Polymers

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. When handling chemicals, you must wear your ( ).
    b. Food coloring may ( ) your clothes or hands.
2. The PVA and borax polymer that you will make in this lab is an example of what type of polymer?
3. Give two examples of amorphous polymers.

Experiment 10: Making Polymers

Experiment Procedure:

Making Glue Solution: Open your test tube that contains the Elmer's glue. Add warm water to the 50 mL mark on the side of the tube. Cap the test tube and shake it well.

Making Borax Solution: Pour all of your borax (in a Ziploc bag) into a small jar. Add ½ cup of warm water and stir it up with a plastic spatula. Make sure all the borax powder dissolves before use.

Making Polymers: In a small Ziploc bag, place 2 tbsp of your glue solution and ½ tsp of your borax solution. Add food coloring if you want to make it look good (Caution: Food coloring may stain your hands or clothes). Mix or smush the polymer in your Ziploc bag and knead it for SEVERAL minutes. **Take a picture for your photo logbook once it forms a ball.** Slowly pull your polymer and record your observations. Abruptly pull polymer and record your observations. Roll the polymer into a ball and drop it. Record your observations. Repeat your experiment again; but this time, add 1 tbsp of cornstarch to the mixture. **Take a photo of the polymer ball** and record all your observations.

Waste Disposal: The jar should be washed thoroughly with soap and water. The polymer balls can be thrown away in the trash can. Any excess glue or borax solution can be pour down the sink with copious amounts of water. If you get some of the polymer solution on your hands, it should come off with soap and water.

Experiment 10: Making Polymers

Photo Logbook **Name:**

1. Insert a picture of your polymer ball (no cornstarch).

2. Insert a picture of your polymer ball (cornstarch added).

Experiment 10: Making Polymers

Post-Lab Questions **Name:**

1. Polymer Observations: Write down your observations for what happens in each case.

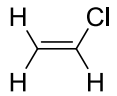
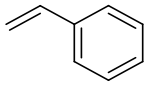
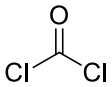
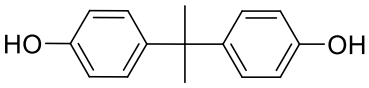
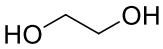
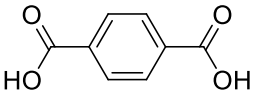
Polymer Sample	Amount of Cornstarch	Slowly pull	Quickly pull	Dropped
1				
2				

2. Which of your polymer balls had the most bounce from the drop test? What conclusions can you draw relating your ingredients to the amount of bounce you observed?

3. Match the following polymer with the common product in which it is used (see intro for help).

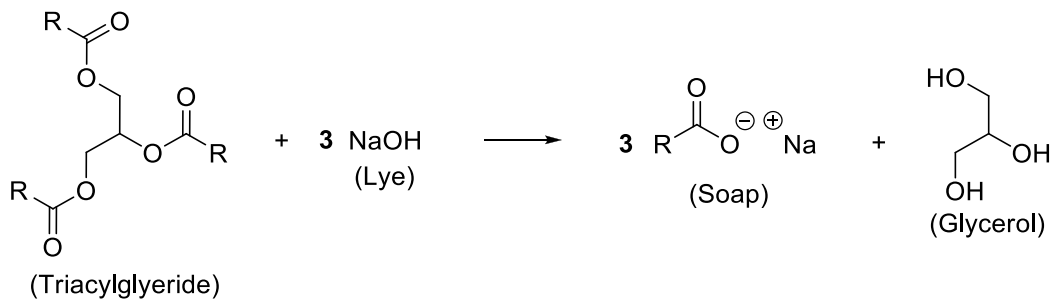
_____ 1. polystyrene	a. Plastic Bags
_____ 2. nylon	b. Water Pipes
_____ 3. polypropylene	c. Cookware
_____ 4. Low density polyethylene	d. egg containers
_____ 5. polycarbonate	e. milk cartons
_____ 6. polyethylene	f. Carpet and Knitted fabrics
_____ 7. polyvinylchloride	g. automobile bumpers
_____ 8. polytetrafluoroethylene	h. Rope

4. Shown below are monomers for common polymers that get made. Use Chemsketch to draw two repeating units of the polymer structure for each one. Use R-groups to show the begin and end of the polymer when applicable.

Type of Polymerization	Monomer(s)	Repeating polymer unit (2 units)
Chain Growth	 vinyl chloride	polyvinyl chloride
Chain Growth	 styrene	polystyrene
Step Growth	 phosgene  bisphenol A	+ HCl is released as other product. This is Lexan polycarbonate (used for bulletproof windows).
Step Growth	 ethylene glycol  terephthalic acid	+ H_2O is released as other product. This is polyester when used for clothing or PET when used for water bottles.

Experiment 11: Preparation of Soap

Soaps are alkali metal salts of fatty acids. They can be created by basic hydrolysis (saponification) of a fat or oil. Fats and oils are known as triacylglycerides and contain three ester functional groups that link a glycerol molecule to three fatty acids. The three ester groups can be hydrolyzed using lye (sodium hydroxide) to form glycerol and soap.



The length and degree of unsaturation in the hydrocarbon chain (R-group) determines the characteristics of the soap. Soaps made from coconut oil contain high amounts of sodium laurate (sodium salt of lauric acid) which is very bubbly and hard. Soaps made from olive oil contain large amounts of sodium oleate (sodium salt of oleic acid) which has skin moisturizing properties.

In this experiment, you will create a bar of soap. The oil mixture that you will be using contains olive oil, coconut oil and lard. The lard and coconut oil will make the soap hard and good for cleaning while the olive oil will produce a soap that is not too drying to skin.

Materials

250-mL plastic beaker
Plastic spatula
Small cooking pot
One 15-mL test tube
Stove
Bowl of hot water
Plastic pipet
Butter knife

Chemicals

Oil mixture (in jar)
Lye solution (in test tube)
Red cabbage indicator solution
Water

Safety: Make sure to wear your safety goggles when handling any chemicals. You will be using a stove so be careful and don't burn yourself. Wear latex, nitrile or rubber gloves when handling the lye solution; it will burn if it comes in contact with skin. If you get some of the lye solution on your skin, flush the area with large amounts of cold water.

Experiment 11: Preparation of Soap

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses to answer the following questions:
    a. If you get some of the lye solution on your skin, you should ( ).
    b. Make sure you wear your ( ) when handling chemicals.
2. Soaps made from olive oil have what kind of properties?
3. If your hardened soap is higher than a pH of 10, what should you do with it?

Experiment 11: Preparation of Soap

Experiment Procedure

Preparing Soap: Pour the oil mixture into a small cooking pot. Lightly heat the mixture on the stove (turn the dial to LOW-MED). You'll notice that the cloudy oil becomes transparent as it heats. While the oil is heating up, place the test tube contain the lye solution in a bowl of hot water. DO NOT TAKE THE CAP OFF. You want use the hot water bath to heat up the lye solution inside the test tube. Once the oil mixture has become transparent pour it into the 250-mL beaker. Let the oil mixture cool down. You want the lye solution and the oil mixture to be about the same temperature (warm to the touch). Once both solutions are about the same temperature, uncap the lye solution and pour it into the oil mixture in the beaker. Immediately after pouring the lye solution in, you'll notice that there are two separate layers of liquid. Use the plastic spatula to stir the mixture. You want the two layers to mix. As you mix, the solution will become cloudy. Stir the mixture periodically (every few minutes) for two hours. DO NOT LET THE MIXTURE SEPARATE INTO TWO LIQUID LAYERS. Over the two hour period, the solution will begin to thicken and the yellow color will start to lighten. It will have about the consistency of lotion. **Take a picture of your oil/lye mixture after you have stirred it for two hours.** After the two hours of mixing, let the mixture sit, undisturbed for 48 hours. The solution should harden over the 2-day period. **Take a picture of your hardened soap.**

With a clean plastic spatula, take about a pea-sized amount of your soap and put it into a 15-mL test tube. Fill the test tube about half full of water and mix the soap thoroughly in the test tube. Using a plastic pipet, add 25-30 drops of your red cabbage indicator solution (from the pH experiment). Mix the solution well and determine the pH based on the color of the solution. **Take a picture of the colored solution after you added the red cabbage indicator.** If the pH is less than 10, it is safe to use. If the pH is greater than 10, the soap could be irritating to skin and should be disposed of. If the soap is safe to use, you can cut it into bars or disks using a butter knife.

Waste Disposal: Test tubes and all plasticware should be washed thoroughly with soap and water, dried then placed back in the chemical/equipment kit. The soap can be thrown away in the trash can (or you can keep it and use it if it's not too basic).

Experiment 11: Preparation of Soap

Photo Logbook **Name:**

1. Insert a picture of the oil/lye mixture after you have stirred it for 2 hours.

2. Insert a picture of the hardened soap.

3. Insert a picture of the colored solution after you added the red cabbage indicator.

Experiment 11: Preparation of Soap

Post-Lab Questions

1. What was the color and pH of your soap solution according to the red cabbage indicator solution?

2. Use Chemsketch to draw the structure of sodium laurate.

3. Use Chemsketch to draw the structure of sodium linoleate.

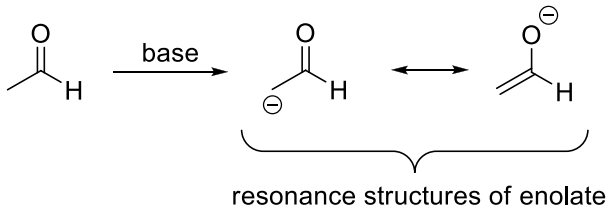
4. A soap made of mostly of sodium laurate will harden in about a day but a soap made mostly of sodium linoleate takes much longer to harden. Why?

5. Sodium acetate and sodium propionate make poor soaps. Why?

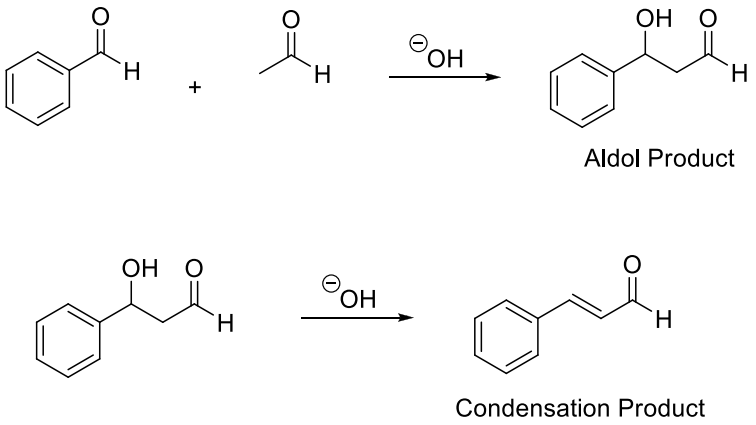
6. What is soap scum and what causes it?

Experiment 12: Crossed Aldol Condensation

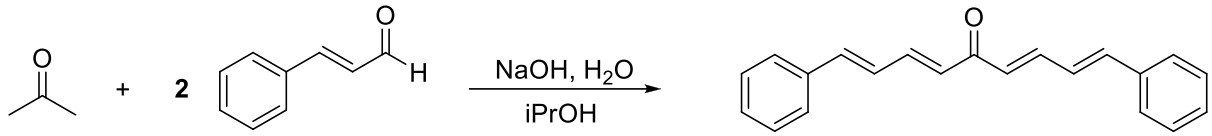
Carbons next to carbonyls are known as alpha carbons (α carbons). Hydrogens on α carbons can be slightly acidic (pK_a ~19-20). Upon treatment with base, hydrogen-containing α carbons can be deprotonated. The deprotonated species is called an enolate and has resonance structures.



Enolates can attack other carbonyl containing compounds to form a new carbon-carbon bond. The crossed aldol reaction is a type of reaction in which two different carbonyl-containing compounds react to form a new larger carbonyl compound. Shown below is the reaction of acetaldehyde with benzaldehyde in the presence of base. The first product that is formed is known as the aldol product (name comes from the **ald**ehyde-alcoh**ol** that is formed in the reaction). If water is eliminated from the aldol product, an α,β-unsaturated carbonyl will be formed. This product is known as the condensation product.



In this experiment, acetone and cinnamaldehyde will undergo <u>two</u> aldol condensation reactions to form a highly conjugated ketone.



Materials

One 50-mL test tube
Two coffee filters
Jar
bottle)
Two plastic syringes
Funnel
Bowl of ice
Plastic spatula
Paper towels

Chemicals

Cinnamaldehyde (in vial)
Acetone (in vial)
50% NaOH solution (in dropper

91% isopropanol
Ice water

Safety: Make sure to wear your safety goggles when handling any chemicals. Wear latex, nitrile or rubber gloves when handling the 50% NaOH solution; it will burn if it comes in contact with skin. If you get some of the NaOH solution on your skin, flush the area with large amounts of cold water.

Experiment 12: Crossed Aldol Condensation

Pre-lab Exercise **Name:**

This Pre-lab must be completed and emailed to the CH 2501 email account (your email here) BEFORE you begin the experiment. Failure to submit this document prior to your worksheet (as documented by the email time stamp) will result in a grade of "0" for your photo logbook and post-lab questions.

Pre-lab Questions:

1. Safety: Read the safety section of your experiment and fill in the blanks between parentheses or answer the following questions:
    a. Make sure to wear your ( ) when handling chemicals.
    b. If 50% NaOH comes into contact with your skin, what should you do?
    c. How will you protect your hands from the 50% NaOH solution?
2. Why is the first product of this reaction described as an "aldol"?
3. What are the two organic reactants you will use for your experiment?

Experiment 12: Crossed Aldol Condensation

Experiment Procedure

Crossed Aldol Reaction: Fill a 50-mL test tube with 5 mL of 91% isopropanol. Add 3.5 mL of cinnamaldehyde and 1.0 mL of acetone using two different plastic syringes. Swirl the mixture in the test tube. To the solution in the test tube, add 10 drops of 50% NaOH. Cap the test tube and swirl the mixture vigorously for 10 minutes. **Take a few pictures of the test tube over the 10 minute period.** Place the test tube in a bowl of ice and let it sit for 5 minutes. While the reaction is cooling, fold the two coffee filters in half (twice) then place it inside the funnel. Put the funnel on top of the jar and filter the cool reaction mixture (see picture below). If the solid sticks to the test tube, pour some ice water into the test tube and use the plastic spatula to break up the solid. Try to transfer as much solid as possible to the funnel for filtration. Wash the solid in the funnel with small amounts of ice water. Allow the mixture to filter until no more liquid is visible dripping from the funnel. Carefully lift the filter paper out of the funnel and place it on a few paper towels. Flatten the solid out on the filter paper and let it air dry for a few hours. **Take a picture of your final product.**

Waste Disposal: Test tubes, funnels and all other glassware/plasticware should be washed thoroughly with soap and water, dried then placed back in the chemical/equipment kit. The liquid obtained from the filtration can be washed down the sink with copious amounts of water. The solid and filter paper can be thrown away in the trash can. If the solid residue in the test tube doesn't come out, use some 91% isopropanol to wash it out.

Experiment 12: Crossed Aldol Condensation

Photo Logbook **Name:**

1. Insert a few pictures of the reaction taking place over the 10 minute period.

2. Insert a picture of your final product after it has dried.

Experiment 12: Crossed Aldol Condensation

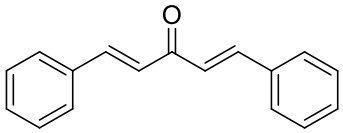
Post-Lab Questions **Name:**

1. Describe what took place during the 10 minutes reaction time (color changes, solid formation, etc.).

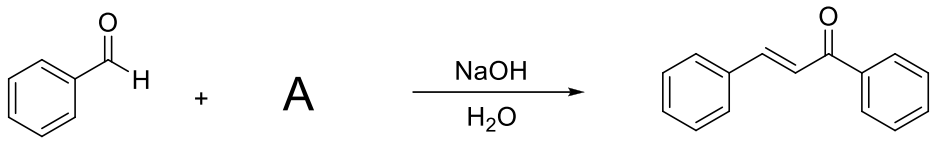
2. What color is the solid product?

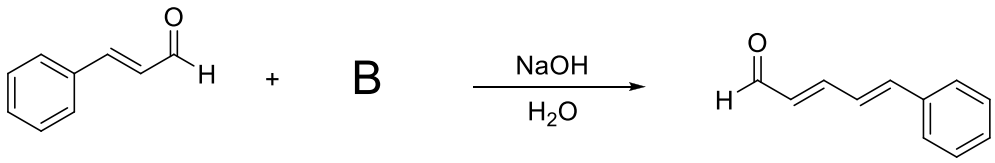
3. Calculate the molar mass of acetone (C_3H_6O) and cinnamaldehyde (C_9H_8O). Show the calculation.

4. How would you synthesize the following compound using an aldol condensation reaction similar to the one used in this experiment? Use Chemsketch to show the reactants and reagents that you would need to synthesize the following product.



5. What is the missing reactant in the following aldol condensation reactions? Use Chemsketch to draw structures A and B.





6. In the experiment that you performed, two molecules of cinnamaldehyde reacted with one molecule of acetone to produce the product that you isolated. What would be the product if one molecule of cinnamaldehyde reacted with one molecule of acetone in an aldol condensation reaction? Use Chemsketch to draw the structure of this product.