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Synthesis of the Moderately Basic Soap Utilizing Fatty Acid Waste

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Abstract Cost-effective and green chemistry methods are appealing in both teaching and research laboratories. We present how fatty acid waste mixtures can be recycled in a subsequent soap-making experiment. Colligative Properties experiments that utilize fatty acids as both solute and solvent in General Chemistry 2 lab courses work well. However, the drawback of this experiment is the laborious clean-up associated with fatty acids adhering to the glassware. Fatty acids, or mixtures thereof, are also used as reactants in soap-making experiments. We recycle the fatty acid mixture in a separate soap synthesis reaction. The methodology adopted in this approach reduces both chemical waste and, more importantly, promotes greener methods in teaching undergraduate chemistry laboratories.

Keywords Soap, Colligative Properties, Freezing Point Depression, Melting Point, Fatty Acids, Green Chemistry, Vernier LabQuest

1. Introduction

Ideal experiments in undergraduate teaching labs utilize nonhazardous inexpensive reagents that generate minimal waste. The experiments selected in teaching labs need to illustrate the course content well with a sure outcome, even if the students deviate from the prescribed procedure. We can tie together two experiments that function very well as illustrations so that the second experiment remedies the difficult clean-up of the first experiment.

A main topic in the second semester general chemistry course is colligative properties, looking at changes in vapour pressure, melting point, boiling point, or osmotic pressure. Changes in vapour pressure or osmotic pressure measurements are more challenging to complete than experiments involving melting or boiling point change. Saltwater solutions are commonly used as they are non-hazardous, easy to rinse and also include experience with the van't Hoff factor [1-4]. However, it does require large quantities of salt, leading to a salty residue that can be found on counters for weeks to come. In addition, problems with forming a supercooled liquid in a freezing point depression experiment can frustrate both student and instructor.

A colligative properties experiment involving fatty acids as solute and solvent remains a simple, nonhazardous option. [3] Using stearic acid as a solvent, students can determine its melting point via a temperature probe and graphical analysis when a sample is merely heated in a hot water bath. After adding an unknown solute (palmitic, myristic, or lauric acids) to stearic acid solvent, students melt the mixture and use graphical analysis to determine the mixture's melting point. The change in melting point is used to identify the unknown solute. If graphs are overlaid, students are able to visually see the depression of the melting point, something that helps solidify the freezing point depression concept in their minds.

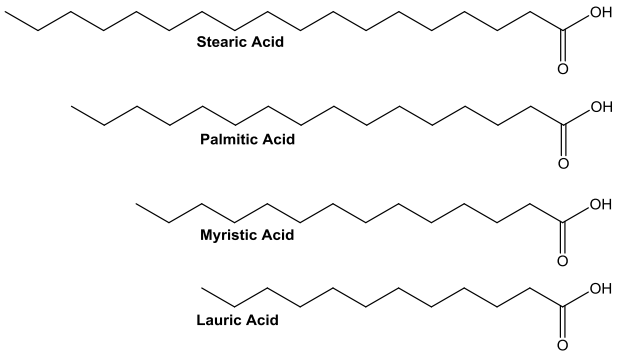

Figure 1. Fatty acids selected for Colligative Properties experiment

While this colligative properties experiment works very well, it leaves much to be desired for student clean-up. The waxy residue in the glassware is nearly impossible to remove completely, even with melting and/or soap. However, the waste generated in this experiment is a fatty acid mixture, a reactant needed in a typical soap formation experiment. Second-semester general chemistry does not cover saponification. However, organic chemistry-related laboratory courses include saponification experiments [5-12]. The nonhazardous waste generated from the freezing point

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depression lab can be used as a reactant in the soap-making lab.

The saponification reaction has been known since ancient times. Our ancestors used the saponification reaction to produce the soap. The long hydrophobic carbon chain of the soap molecules is soluble in nonpolar solvents, while the carboxylate group is soluble in polar solvents like water. Due to these features, soap molecules are capable of forming micelles. The hydrophobic center of the micelle allows nonpolar substances to easily dissolve. At the same time, the micelle itself is water soluble. The completion of the soap formation reaction using the fatty acid mixture from the colligative properties experiment ensures the easy clean-up of the glassware.

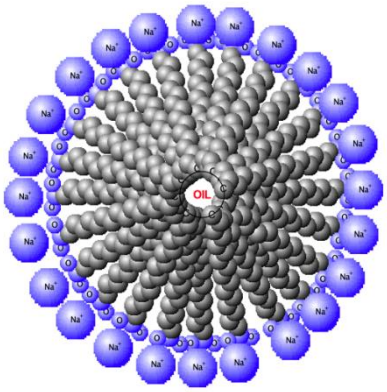

Figure 2. Formation of the soap micelle with the oil being in the center of the micelle

Over the past several decades, many reports have been published on soap synthesis [5-13]. The major drawback in the synthesis of soaps outlined in many reports is the wait time necessary for the soap to cure [5-12]. Typically, soap produced in saponification reactions has a pH above 12. Students typically cannot handle the soap without protective equipment. Thus, the soap is either left to cure for several weeks or partially neutralized in an acid-base reaction. The pH of the synthesized soap could be reduced by natural acid sources (apple juice, lemon juice, lime juice, and cranberry juice), allowing students to immediately test the properties of soaps instead of waiting for soap to cure.

2. Experimental Part

Materials needed: Supplied in the supplemental part of the manuscript.

2.1. Colligative Properties Experiment

Though unnecessarily large for this portion of the experiment, students should obtain approximately 8 grams of stearic acid in a pre-weighed 150mL beaker. The size of the beaker is based on the needed reagents for the recycling of the product in the subsequent soap synthesis experiment. The amount of stearic acid need not be precise at this time; however, the mass of the empty beaker must be known.

The beaker containing the stearic acid should be suspended in a hot water bath using a 400mL beaker or larger on a hot plate. (Figure 3a). The water should be allowed to warm above 80°C, but not boil, as condensation from the water vapours will skew calculations. Once the stearic acid melts (above 69.3°C), the resulting liquid should be allowed to warm slightly. After that, remove the beaker from the warm water bath. With constant stirring, the fatty acid mixture will solidify. During this process, students record the temperature at consistent intervals using LabQuests with temperature probes or a comparable product. After the stearic acid freezes, the temperature will drop, and students can stop recording data. Students can determine the melting point of the stearic acid using the average value found from the shaded constant temperature region of their graph (Figure 3b).

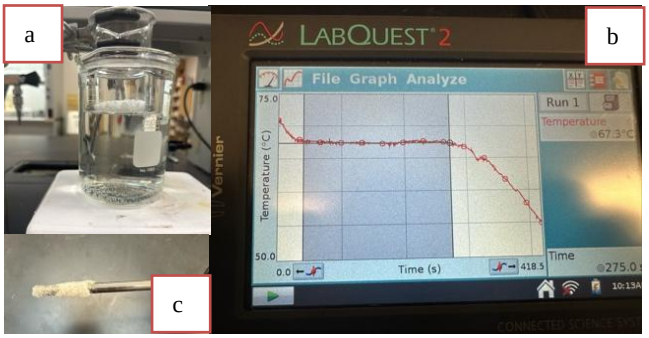

Figure 3. a) Colligative Properties setup using the fatty acid mixture. b) Temperature vs. time graph. c) Adherence of fatty acid to the probe

The stearic acid will adhere to the temperature probe as it solidifies. (Figure 3b). If possible, the students should scrape off the stearic acid into the beaker. If not, students can use a paper towel to wipe off the temperature probe and discard it. At this time, students should weigh the beaker and determine how much stearic acid remains in the beaker.

Students are assigned an unknown component of either palmitic, myristic, or lauric acid, and should add approximately 1 gram to their beaker. It is necessary to know the amount of unknown added to the beaker precisely. Therefore, students are encouraged to add it directly to the beaker instead of a weigh boat, even if it means too much unknown acid is added.

Students reheat the mixture in the hot water bath as before. The mixture may require them to heat the contents above the melting point of stearic acid to make a homogeneous solution. Similarly, students remove the beaker from the hot water bath and collect the temperature data at constant intervals with continuous stirring. Students will only need to collect data until the mixture solidifies and the temperature graph's slope changes. The point at which the slope changes is the melting point of the mixture. Students should be encouraged to do this step more than once, as slight variations in this measurement will significantly impact the calculated molar mass.

When the experiment is complete, the stearic acid mixture clings to the glassware. Students should write on the side of

the beaker the mass of the empty beaker for use in the soap-making experiment.

2.2. Soap Synthesis Experiment

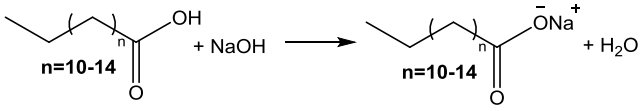

Figure 4. Soap Formation Equation

Boil approximately 200 mL of tap water in a 600 mL beaker on the hot plate. It is prudent to confirm the mass of the fatty acid mixture present in the starting beaker by subtraction. Heat the fatty acid mixture (Figure 5a) with 20 mL of ethanol in the water bath. The slow addition of 5 mL of 6M NaOH solution promotes the dissolution of the fatty acids in ethanol.

The mixture will need to heat in the hot water bath with periodic stirring for approximately twenty minutes. The disappearance of the ethanol scent signifies the completion of the reaction. Precipitation of soap occurs as the mixture cools to room temperature. The formed soap chunks are collected via vacuum filtration using filter paper and rinsed with cold water. (Figure 5b). The pH of the soap is expected to be over 12, making it highly caustic and unsafe to use. Students neutralize half of the soap product to a pH of 8 by addition of fruit juice while leaving the other half to cure for two to three weeks. (Figure 5b and 5c). The fruit juice was added dropwise with frequent checks of pH until the desired pH =8 value was reached. The typical choices of juices were apple juice concentrate, lemon, and lime juices. The fruity soap can be safely handled and tested immediately. The untreated soap can also be tested if desired with appropriate safety attire. Students compare the foaming ability of the soaps by mixing a few drops of the mineral oil placed into test tubes with equal amounts of the uncured sample of treated and untreated synthesized soap versus commercially available soap. The formed soap has the consistency of powdered detergents, unlike traditional saponification experiments using fats or oils. The successful formation of the soap was confirmed by melting point determination. The anticipated melting points of soap products exceed 230℃.

3. Results and Discussion

3.1. Colligative Properties

The data collected should be saved during the experiment. If the experimentally determined freezing point is not as expected (69.3℃, within the expected error of the machine), it is likely that the students did not select the appropriate portion of the pure stearic acid graph (Figure 3b). Students also have difficulty determining the melting point of the mixture. If multiple trials show inconsistent results, students should revisit the graph data and extrapolate straight lines for the slopes before and after freezing. The intersection of these lines will provide a more accurate measurement of the mixture's freezing point. Overlapping all the graphical data provides a visual representation of freezing point depression. The sample data is provided in the supplementary part of this manuscript. Students should have recorded the melting point of both pure stearic acid and the mixture. They will need to calculate the mass of stearic acid remaining in the beaker and the mass of unknown acid added to the beaker. The molality of the mixture can be found from the change in melting point and the known K_f value of stearic acid (4.5℃/*m*). After the conversion of the mass of solvent (stearic acid) to kilograms, students can determine the moles of solute and, subsequently, the molar mass. The calculated molar mass typically only has about two significant figures as the change in melting point is limited by the precision of the temperature probe. Students should be reminded when identifying the unknown acid. Students must write the mass of the empty beaker on the side before finishing the experiment, as the information is necessary and helpful for the subsequent soap making reaction.

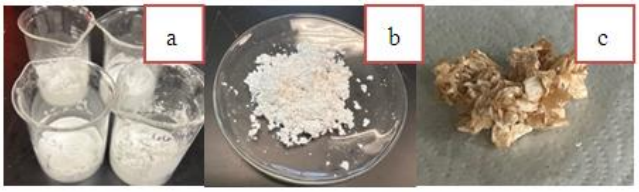

Figure 5. a) Fatty acid mixtures after the "colligative properties "experiment. b) Soap product after soap synthesis reaction. c) Soap product after soap synthesis reaction neutralized with fruit juice

3.2. Synthesis of Soap

The fatty acid waste consisted of a mixture of Stearic acid (major component) with either lauric, myristic, or palmitic acid (minor components). Students determine the melting points of the fatty acid mixtures prior to soap-making, which are in the expected broad range of 60-70℃. The formed soap can be characterized as a white powdered substance with no distinct scent and poor water solubility. The pH tests confirmed that the formed soap's pH exceeded the pH of 12. Although students were not expected to calculate the exact product yield, we expected reaction to yield soap quantitatively. The melting point of the synthesized soap samples was reported to be in the range of 230-260℃ supporting the formation of the product.

4. Conclusions

Designing undergraduate chemistry lab experiments that produce low to no waste remains one of the goals of teaching undergraduate chemistry. It also aligns with the twelve principles of green chemistry. In the pairing of a general chemistry colligative properties lab with an organic chemistry soap making lab, we are able to reduce waste and ease student clean-up while still retaining student laboratory experiences. The colligative properties experiment effectively demonstrates the freezing point depression of stearic acid due to the addition of either palmitic, myristic or

lauric acid. The resulting waxy fatty acid readily reacts with sodium hydroxide in a reaction generating sodium stearate mixed with an additional sodium salt, confirmed via melting point. While the fruit juice concentrates lower the pH, it limits the efficacy of the resulting soap. Upon neutralization or curing of the sodium stearate mixture, the nonhazardous materials are effectively used twice, lowering the cost of materials.

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Supplementary Information

The complete lab handouts can be accessed using the following link: will be provided later in the journal upon acceptance of manuscript.

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