The Soapmaking Factory Lab

Overview
In this lesson students will create a portfolio of environmentally friendly soaps with different properties, shapes, colors and aromas. Students will learn about the chemical reactions necessary to create soaps. Students will formulate their soaps by combining different oils (e.g., olive, palm, and coconut oil) and essential oils to explore various qualities of soap including antibacterial capacity, foamability, detergency, oiliness on the skin, spreading, scent, appearance, and consistency. Student will compete to see who produced the most balanced and creative soap portfolio.

Content Standards
This lesson is appropriate for earth/environmental science, biology, physical science, and chemistry students, and it addresses the following standards:

North Carolina Essential Standards

Earth/Environmental	EEn.2.2.1; EEn.2.6.3; EEn.2.8.2
Biology	Bio.2.2.1, Bio.2.2.2, Bio.4.1.1
Physical Science	PSc.2.1.3, PSc.2.2.2
Chemistry	Chm.1.2.3, Chm.2.2.2

Next Generation Science Standards
Grades 9-12, Science and Engineering Practices and Cross-cutting Concepts:

Science and Engineering Practices	Asking Questions and Defining Problems; Planning and Carrying Out Investigations; Analyzing and Interpreting Data; Constructing Explanations and Designing Solutions
Cross-Cutting Concepts	Patterns; Cause and Effect: Mechanism and Prediction; Structure and Function

Time Requirements
Preparation: 30 minutes
Class-time: 2 class periods. The first period will cover the soap preparation, and the second period the testing of the soap performance and properties.

Materials
Included in the kit:
Thermometer

Hot plate
Silicone soap molds
Glass beaker (2000 mL)
Glass beaker (600 mL)
Glass beaker (250 mL)
Glass rod
1-L graduated cylinder
Separation funnel
Stainless steel or silicone egg whisk
pH test paper
Glo-germ experiment kit (lotion and powder)
Petri dish
Gloves
Safety goggles
Lab coat
Essential oils (optional)
Soap dyes (optional)
Sodium hydroxide in pellets
Unrefined virgin coconut oil
Palm oil
Olive oil

Needed, but not supplied:
Precision balance (readability = 0.02 g)
Distilled water

Safety

Ensure that students understand and adhere to safe laboratory practices when performing any activity in the classroom or lab. Demonstrate the protocol for correctly using the instruments and materials necessary to complete the activities, and emphasize the importance of proper usage. Use personal protective equipment such as safety glasses or goggles, gloves, and aprons when appropriate. The use of gloves through the laboratory is highly recommended. Model proper laboratory safety practices for your students and require them to adhere to all laboratory safety rules.

Sodium hydroxide will burn skin and eyes. Do not touch sodium hydroxide in any form with bare hands. In case sodium hydroxide is splashed onto the skin, use vinegar and plenty of water to wash immediately. If it is splashed into eyes, use plenty of water to flush them for at least 15 minutes, and contact a physician. If sodium hydroxide gets on any clothing, remove the clothing and wash prior to reuse.

When combining sodium hydroxide (lye) with water, always add the lye slowly to cool water while stirring carefully. The medium will heat up. Use gloves to hold the beaker where the sodium hydroxide is prepared. The sodium hydroxide solution should not be splashed while stirring. Whenever sodium hydroxide is dissolved in water, vapors are released. Do not breath these vapors as they contain molecules of sodium hydroxide.

Prepare the solution in a well-ventilated area (use of a fume hood is recommended).

Beware that laboratory hot plates present dangers such as the potential for people to burn themselves or even start a fire. Remember to turn the hot plate off once you finish working with it. Do not overheat the polymer melt to avoid potential spilling of hot components. Be extremely careful when manipulating the polymer melt during the formation of the film.

Background Information

1. Soap making: saponification reaction

Historically, soap has been a key product in people's personal hygiene routine. Ancient Egyptians used a substance very similar to our modern soap 4,800 years ago. Especially nowadays, when the rise of hygiene standards has become critical, the use of soap is more essential than ever.

Soap is both environmentally friendly and biodegradable, given its natural origins. Soap is made by boiling fats, either vegetable (fatty acids) or animal (tallow), in a strong alkaline solution. Such a reaction is called saponification (Fig. 1). Fats are triglycerides, composed of three fatty acids attached to a 3-carbon glycerol backbone. The bond between the fatty acid and the glycerol backbone is referred to as an ester linkage. In the saponification process, the ester linkage is broken by a strong base, such as sodium hydroxide (NaOH), also called lye, to form glycerol and a fatty-acid salt, or soap.

In soap making, the essential reaction occurs in two stages. The first stage is the hydrolysis, where the three fatty acids of each triglyceride are removed from the glycerol backbone, resulting in one molecule of glycerin and three fatty acid molecules. The driving force for this reaction is the raise in pH caused by the strong base (or alkali), which makes the pH of the medium alkaline.

$$Triglyceride + Lye\ solution \rightarrow 3\ Fatty\ acids + Glycerin$$

The second stage is the saponification, where the strong base reacts with each fatty acid, replacing the hydrogen of the acid with the sodium (Na^+) or potassium (K^+) in case potassium hydroxide (KOH) is used as the strong base, to produce a salt. This salt is the soap. This reaction does not alter the glycerin, which can be separated from the mixture or left *in situ* to enhance the creamy texture of the resultant soap.

$$Fatty\ acid + NaOH\ (Lye) \rightarrow Soap + Water$$

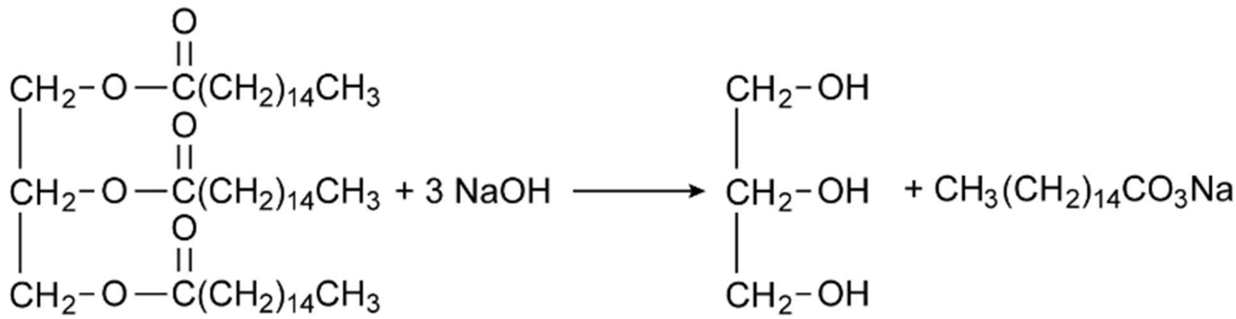


Fig. 1. Saponification of a triglyceride with sodium hydroxide to form a soap

The selection of the fatty acid to undergo saponification will depend on the soap use. Fig. 2 shows the fatty acid composition of some common triglycerides used in soap making. Typically, luxury soap bars such as Palmolive are made only with vegetable oils having a composition of C16-C18, such as palm and olive oils. These soaps do not produce skin irritation but tend to produce white deposits when exposed to hard water (also known as calcium saps) due to poor solubilization. In that sense, C12-C14 soaps such as the ones produced from coconut oil, are typically added in small proportions (about 25%) to increase foamability and tolerance to divalent cations (Ca^{2+} and Mg^{2+}). Tallow (beef fat) can also be used to produce C16-C18 soaps with a larger proportion of unsaturated C18 but at a cheaper cost. Mixtures of tallow with vegetable oils can also be used to obtain a variety of soaps exhibiting different attributes.

Soaps made with sodium hydroxide (NaOH) are not harmful to the skin if the soap is made correctly. If too much NaOH is used for the fat content present in the reaction medium, excess NaOH may remain unreacted. In order to neutralize the NaOH's caustic properties, it is possible to "super fat" the soap, this is to reduce the amount of NaOH required for complete saponification so that an excess of unreacted triglycerides remain in the finished soap.

ACID ↓ / OIL / FAT →		COCONUT	ALMOND	PEANUT	SOYA	OLIVE	CORN	PALM	PORK FAT	BEEF FAT	BUTTER
Caprilic	C08:0	07	04	-	-	-	-	-	-	-	01
Capric	C10:0	08	04	-	-	-	-	-	-	-	03
Lauric	C12:0	48	50	-	-	-	-	-	-	-	04
Myristic	C14:0	17	16	-	-	-	-	01	01	02	12
Palmitic	C16:0	09	08	11	11	14	12	46	26	35	29
Stearic	C18:0	02	02	03	04	03	02	04	11	16	11
Oleic	C18:1	06	12	46	25	68	27	38	49	44	25
Linoleic	C18:2	03	03	31	59	13	57	10	12	02	02
Linolenic	C18:3	-	-	02	08	-	01	-	01	-	-

Beef fat = tallow

Fig. 2. Fatty acid composition (%) of some triglycerides

2. How soap interacts with dirt: washing mechanism

Soap is composed of molecules having a polar-apolar duality (Fig. 3). The polar part is "hydrophilic" (or water-loving), and thus exhibits a strong affinity toward polar solvents, such as water. On the other hand, the apolar part is "hydrophobic" (or water-hating) and is thus attracted toward oils. Soap molecules are schematically represented with a polar head and an apolar tail, which is generally a hydrocarbon fatty chain.

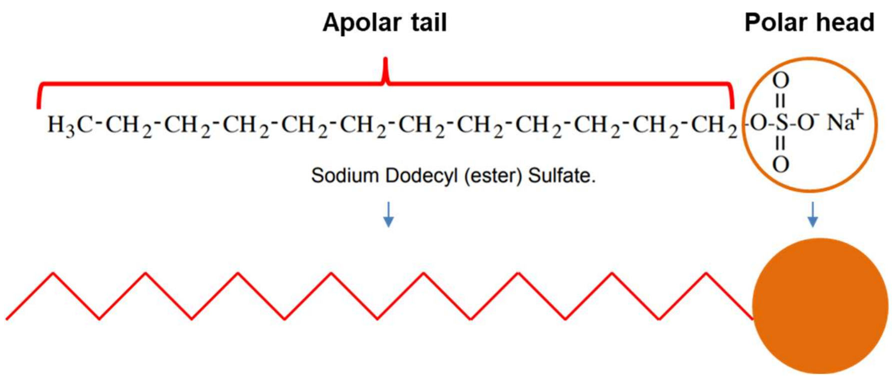


Fig. 3. Soap molecule and schematic representation displaying the polar head and the apolar tail

Fig. 4 shows the mechanism by which soap molecules wash away germs and dirt from the skin. When soap and dirt are mixed, the soap molecules break up the dirt and the bacteria it contains by forming circles around individual droplets. The apolar tails go in the middle facing the dirt, while the polar heads face outwards of the circle facing the surrounding water. This wheel-like structure is known as a micelle. These micelles, which become suspended and distributed in water due to the presence of the polar heads, are then easily rinsed away with water.

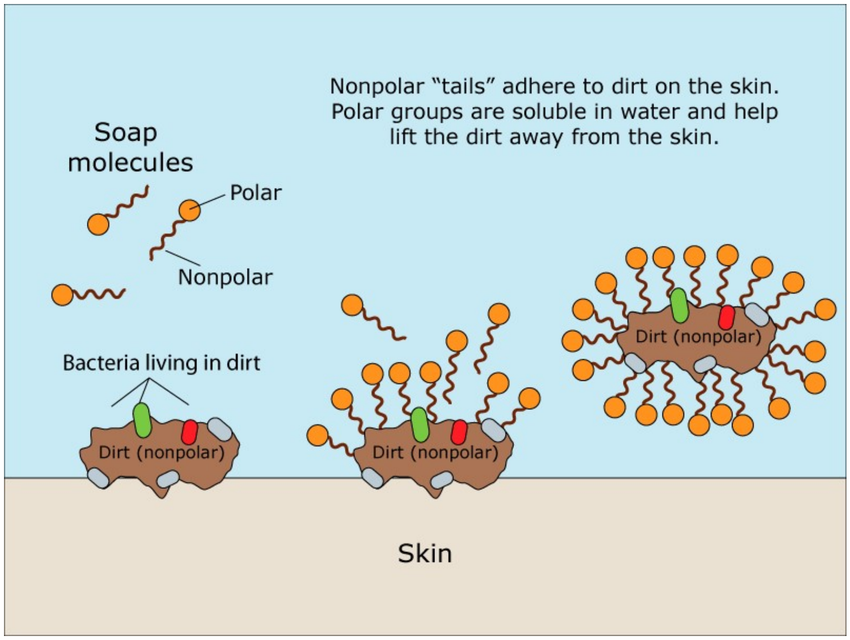
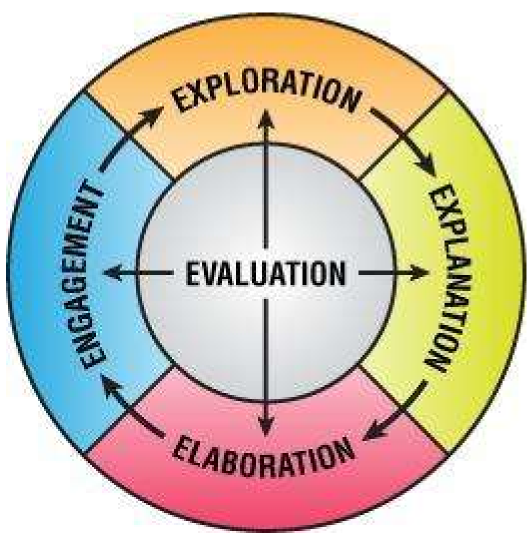


Fig. 4. Mechanism by which soap interacts with dirt present in the skin

Preparation

1. Photocopy the Student Guide for each student or each group, as desired.
2. Organize students into 10 groups of three. If your class size differs, adjust the size of the 10 groups accordingly.
3. Set out the materials for the experiments in a central, accessible location so that students can see them and consider how the might use them. Remember, the method that they pic will determine the basic set of materials they will need.

Guiding the lesson using the 5E Learning Cycle



Engage (10 minutes)
1. Explore with student some of the current events and issues related to oils (deforestation associated with palm oil production, use of cooking oil to create new products).
2. Brainstorming with students on desirable qualities of soap.

Explore (45 minutes)
1. Day 1: Students will formulate soaps with the materials available using the general procedure provided. Use lye calculator to calculate out the amounts of sodium hydroxide, and water needed based on the amounts and types of oil to be used. Teacher floats between groups to ensure student participation and understanding.
2. Day 2 and subsequent: Test the pH of the soap until reaching a value below 10.

Explain (10-15 minutes)
1. Students define and explain the function of each of the components involved in the soap preparation. What factors might influence the process and how?
2. Discuss the chemistry behind soapmaking (saponification reaction).

Elaborate (60 minutes)
1. (5-10 minutes) Start with discussion on what types of properties are important when formulating soaps.
2. Test soap properties indicated in laboratory manual.
3. Discuss group data (create table on whiteboard, or use spreadsheet). Compare groups' results, and talk about any discrepancies between data.

Evaluate (10 minutes)
1. Develop conclusions based on the results. Discuss other tests/ways to evaluate the soap or if students would do anything different if they had the chance to do the soap again. Answer questions in student guide.

Questions in student guide
1. What is the objective of the study?
2. Describe the conditions that you used in your soap making experiment.
3. What results did you see in the experiment?
4. What is the function of each of the components added during the preparation of the soap? What is happening at the molecular level during the preparation of the soap?
5. Why is it necessary to keep the temperature of the mixture at a high value?
6. What does the pH of the soap indicate about the extent of the saponification reaction? Why does the pH change with time?
7. What makes coconut oil especially desired for soap making?
8. What other oils can be used to make soap? What attributes would the selected oils impart to the soap?
9. How can the procedure and materials be modified to produce liquid soap?

10. What would happen if the superfatting level is increased? What are the limits to which this parameter can be modified?
11. How efficient was the soap at removing germs during handwashing? What was the effect of time on germs removal efficiency? What was the minimum time required to get complete germ removal? Where do germs tend to accumulate the most?
12. How efficient was the soap at removing germs from a surface? Were there any major differences when compared with cleaning with just water? How des soap removes dirt?
13. What was the foamability of the soap prepared? How long did it take for the foam to fully break? What is a foam? Was there any difference in the morphology of the foam at the top of the cylinder compared to the foam near to the solution at the bottom of the cylinder? What is the mechanism by which a foam break?
14. What were the results of your sensorial analysis? How would you improve the attributes with the lowest scores? In case that more than one soap was formulated, what is the soap with the highest score?

Supplemental Resources

Soap calculator	https://www.brambleberry.com/calculator?calcType=lye
Coconut oil soap making	https://www.youtube.com/watch?v=5uXbEPy_Xao&t=149s
Surfactants: types and uses	http://nanoparticles.org/pdf/Salager-E300A.pdf
Fun facts about soap	https://www.rompagroup.com/news/17-facts-about-soap-the-most-popular-hygiene-product-in-the-world.aspx
Creative handmade soap recipes for beginners	https://www.thesprucecrafts.com/basic-soap-making-recipes-517179

Name ______________________ **Student Guide**
Date _______________________

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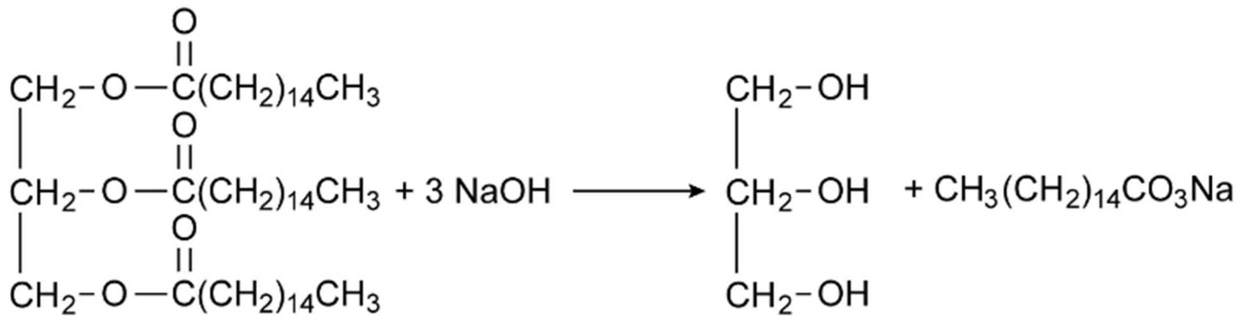

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Myristic	C14:0	17	16	-	-	-	-	01	01	02	12
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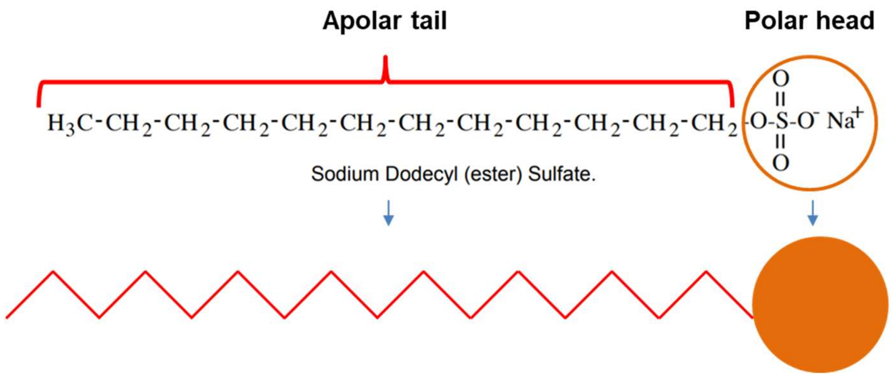
Fig. 2. Fatty acid composition (%) of some triglycerides

2. How soap interacts with dirt: washing mechanism

Soap is composed of molecules having a polar-apolar duality (Fig. 3). The polar part is "hydrophilic" (or water-loving), and thus exhibits a strong affinity toward polar solvents, such as water. On the other hand, the apolar part is "hydrophobic" (or water-hating) and is thus attracted toward oils. Soap molecules are schematically represented with a polar head and an apolar tail, which is generally a hydrocarbon fatty chain.



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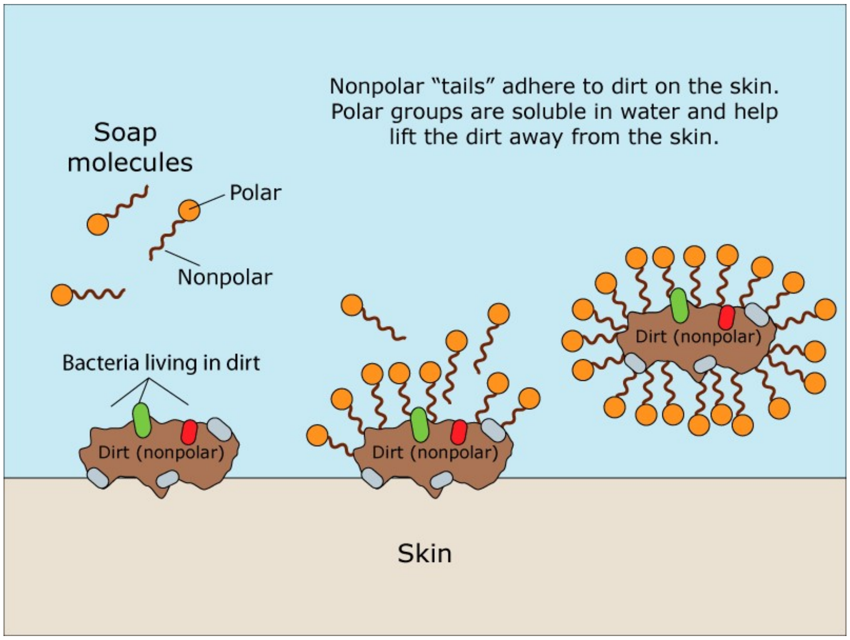

Fig. 4. Mechanism by which soap interacts with dirt present in the skin

Materials

- Thermometer
- Hot plate
- Silicone soap molds
- Glass beaker (2000 mL)
- Glass beaker (600 mL)
- Glass beaker (250 mL)
- Glass rod
- 1-L graduated cylinder
- Separation funnel
- Stainless steel or silicone egg whisk
- pH test paper
- Glo-germ experiment kit (lotion and powder)
- Petri dish
- Gloves
- Safety goggles
- Lab coat
- Distilled water
- Essential oils (optional)
- Soap dyes (optional)
- Sodium hydroxide in pellets
- Unrefined virgin coconut oil
- Palm oil
- Olive oil

Safety

Sodium hydroxide will burn skin and eyes. Do not touch sodium hydroxide in any form with bare hands. In case sodium hydroxide is splashed onto the skin, use vinegar and plenty of water to wash immediately. If it is splashed into eyes, use plenty of water to flush them for at least 15 minutes, and contact a physician. If sodium hydroxide gets on any clothing, remove the clothing and wash prior to reuse.

When combining sodium hydroxide (lye) with water, always add the lye slowly to water while stirring carefully. The medium will heat up. Use gloves to hold the beaker where the sodium hydroxide is prepared. The sodium hydroxide solution should not be splashed while stirring. Whenever sodium hydroxide is dissolved in water, vapors are released. Do not breath these vapors as they contain molecules of sodium hydroxide. Prepare the solution in a well-ventilated area (use of a fume hood is recommended).

Procedure

The quantities indicated below are specified for making soap from coconut oil. In case palm, olive, any other oil or mixtures thereof are used, refer to the lye calculator (https://www.brambleberry.com/calculator) to estimate the amount of NaOH and distilled water needed.

1. **Soap making**
    - 1.1. Weight 300.0 g of coconut oil in a 2000-mL glass beaker
    - 1.2. Weight 48.1 g of NaOH pellets
    - 1.3. Weight 107.5 g of distilled water
    - 1.4. Place the coconut oil on the hot plate and heat it to 85°C. Keep track of the temperature with a thermometer. The coconut oil will melt at around 35°C.
    - 1.5. While heating the coconut oil, prepare a NaOH solution by dissolving the NaOH pellets in the distilled water using the 600-mL glass beaker. Add the NaOH pellets slowly to the water **(always add base to water)**. Stir continuously the solution using a glass rod. Follow the guidelines outlined in the safety section.
    - 1.6. Add slowly the NaOH solution into the coconut oil. Stir gently. One person should slowly pour the NaOH solution to the coconut oil, while other person stirs constantly with the stainless steel egg whisk. Adjust the temperature so that it remains at 85°C.
    - 1.7. Using the egg whisk, continue stirring the mixture until tracing occurs. Tracing refers to the presence of traces of the soap mixture on the surface of the mass when some of the mixture is taken up on the stirrer and dribble back in. This may take up to 30 minutes.
    - 1.8. Add essential oil for scent and coloring (optional).
    - 1.9. Once tracing occurs, quickly transfer the soap to the silicone molds. This should be done quickly to prevent solidification of the mixture.
    - 1.10. Measure the pH of the soap by introducing a pH strip in the mixture. Record this value as pH at day 0.
    - 1.11. Allow soap to harden for about 24 hours before de-molding. After 24 hours de-mold the soap and air-dry it inside a paper bag. Track the evolution of the pH until it reaches a value of 10 or below 10. To measure the pH of the soap bar moist the pH strip with water and rub it over the soap surface.

The soap bar should have a pH between 7 and 10 before it is considered safe to use. This assures that there is no free NaOH remaining in the soap (NaOH has a pH of 14). A pH value above 10, indicates that the soap is harsh, and may have “free NaOH” (NaOH that did not react with the coconut oil and can burn the skin).

2. Testing the soap performance

2.1. Simulated germs removal during handwashing

The objective of this exercise is to determine the time required for total germ removal during handwashing:

2.1.1. Apply simulated germs on palms of hands and rub together for 20 seconds, including backs of hands.
2.1.2. Scrape fingernails on palms to “infect” with “germs” under nails.
2.1.3. Place hands under UV light to view “simulated germs”.
2.1.4. Follow handwashing using soap. Each team member could try to simulate the hand washing strategy that he/she typically follows, or each team member could be assigned different times for applying soap in hands and rinsing it with tap water (for instance, 10 seconds of soap application and 15 seconds for rinsing with water).
2.1.5. Test again with UV light. Take pictures to include in your lab report.

2.2. Simulated germs removal from a surface

The objective of this exercise is to test the efficiency of the soap at removing germs from a surface and compare it to that of pure water:

2.2.1. Sprinkle around 1 g of glow germ powder on the surface of a petri dish.
2.2.2. Spread the powder around the area of the petri dish.
2.2.3. Test with UV light.
2.2.4. Prepare a soap solution by dissolving 1 g of soap in 100 g of water. Soap shavings can be used to facilitate dissolution of soap in water.
2.2.5. Add 50 g of soap solution on petri dish and let it sit for 5 min.
2.2.6. After 5 min gently swirl the petri dish 10 times and then dispose of its content.
2.2.7. Test with UV light. Take pictures to include in your lab report.
2.2.8. Add 50 g of water on petri dish and gently swirl the petri dish 10 times.
2.2.9. Dispose of water and test again with UV light. Repeat the previous procedure using just water and compare your results.

2.3. Foamability test

The objective of this exercise is to measure the foaming capacity (foamability) of a soap by measuring the height of the foam produced from a soap solution made thereof. The following procedure is an adaption of the Ross-Miles method (ASTM D 1173-53):

2.3.1. Prepare 250 g of a 1 wt% soap solution by dissolving 1 g of soap in 250 g of distilled water.

2.3.2. Heat the soap solution to 49°C and then add 50 g to a graduated cylinder and 200 g to a separation funnel.
2.3.3. Measure 1 meter height between the solution contained in the cylinder and the nozzle of the separation funnel.
2.3.4. Place the nozzle in the direction of the cylinder and open the valve completely.
2.3.5. Start a chronometer at the exact moment when the valve opens. Record the initial height of the foam formed. Take measurements of the height first every 30 seconds, and then every 1 min and 15 min after the height changes are not very noticeable. Use the middle of the foam as the reference point to measure the height.
2.3.6. Report the percentage of foam broken as a function of time using as a reference the maximum height reached (Hmax). Hmax represents the foamability of the solution. Calculate Hmax by subtracting the initial height of the foam formed from the 250 mL of the solution remaining in the cylinder once the foam has disappeared. Try to identify differences in morphology (shape and size) of the foam at the top of the cylinder and above the solution at the bottom of the cylinder. Take pictures to include in your lab report.

2.4. **Sensory analysis**

A sensory analysis of the soap is carried out by ranking the following aspects on a scale from 0 to 5 (0 = lowest; 5 = highest):

- Oiliness on the skin: oil reside that is left over the skin after the soap is applied
- Spreading: ease to spread a soap on the skin surface
- Oder or scent: aroma of the soap
- Color: user confidence when observing the soap
- Appearance: granules or impurities observation in the soap
- Consistency: homogeneous or heterogeneous

Questions

15. What is the objective of the study?
16. Describe the conditions that you used in your soap making experiment.
17. What results did you see in the experiment?
18. What is the function of each of the components added during the preparation of the soap? What is happening at the molecular level during the preparation of the soap?
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28. What were the results of your sensorial analysis? How would you improve the attributes with the lowest scores? In case that more than one soap was formulated, what is the soap with the highest score?