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Lab work: industrial organic chemistry
Specialty: Process Engineering
Level: 2nd year LMD

Experiment N°3: Preparation of Soap

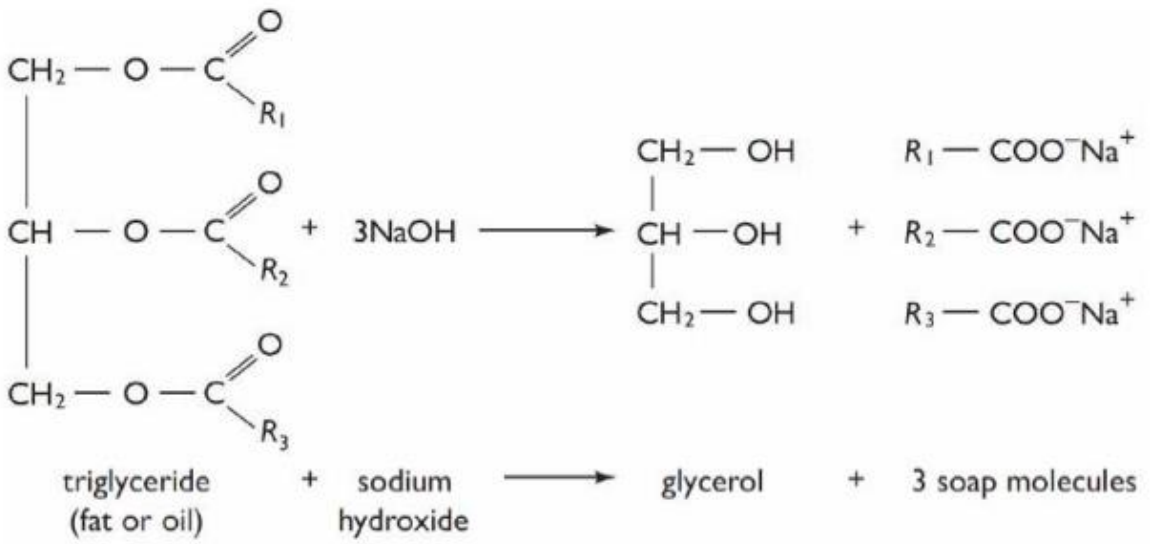
I. Objectives:

1. Understand the saponification process, a chemical reaction between a fatty acid and a base.
2. Produce soap from fats or vegetable oils.
3. Observe and analyse the properties of the obtained soap.

II. Principle:

Soaps are carboxylate salts with very long hydrocarbon chains. it can be made from the base hydrolysis of a fat or an oil. This hydrolysis is called **saponification**.

Saponification is a process in which a fat molecule is broken down by sodium hydroxide (lye) into four smaller molecules; three of the new molecules are soap and one is glycerol. In simple terms saponification is the name for a chemical reaction between an acid and a base to form a salt. An acid is a molecule or ion that donates protons or hydrogen ions (H+) and/or accepts electrons, and a base is any substance that donates electrons or hydroxide ions (OH-) and/or that accepts protons. When you make soap, you mix an oil or fat (which is your acid) with Sodium Hydroxide or Lye (which is your base) to form soap (which is a salt). There are many different types of acids that will react with your base and saponify. Your acid could be olive oil, coconut oil or vegetable oil just to name a few. Each acid has a unique combination of triglycerides (compounds made of three fatty acids attached to a single glycerol molecule) which combines with the base (sodium hydroxide or lye) differently.



III. MANIPULATION :

- **Equipment :**
  - Graduated cylinder
  - Reflux heating apparatus
  - Beaker
  - Hot plate
  - Magnetic stir bar
  - Thermometers
  - Erlenmeyer flask
  - Spatula
  - Electronic balance
  - Wash bottle
  - Vegetable oil (olive, coconut, or other)
- **Reagents**
  - 25 g of vegetable oil (olive, coconut, or other)
  - 10 g of sodium hydroxide (NaOH)
  - 50 mL of distilled water
  - 20 mL of ethanol
  - 10 g of sodium chloride (NaCl) (for soap salting-out)
  - Cold water
  - Colorants and fragrances

- **Experimental Protocol**

1- Preparing the sodium hydroxide (NaOH) solutions:

- Dissolve 10 g de NaOH in 50 mL of distilled water in a beaker.
- Stir with a spatula until completely dissolved. The reaction is exothermic.

2- Heating the oil:

- Place 25 g of vegetable oil in beaker.
- Heat gently in water bath until the temperature reaches 50 – 60°C. Stir with a magnetic stirrer.

3- Mixing the ingredients:

- Gradually add the NaOH and the ethanol solution to the warm oil while stirring continuously.
- Maintain the temperature at 50-60°C and stir for about 30 minutes until a thick emulsion appears (soap formation).

4- Soap Salting-out (relargage):

- Add a saturated NaCl solution (about 10 g in 50 mL of water) o help precipitate the soap.
- The soap will separate as a solid at the surface.

5- Filtration and rinsing:

- Separate the solid soap from the aqueous phase.
- Rinse the soap with cold water to remove any unreacted NaOH.

6- Molding and drying:

- Pour the soap into molds and let it dry in the air for several days.
- Analyze the physical properties (texture, pH, aolubility) of the soap.

IV- QUESTIONS :

1. Provide the diagram of the experimental setup for the synthesis in the laboratory (experimental setup).
2. Write the equation for the saponification reaction.
3. Provide the reaction mechanism.
4. Give the name and formula of the soap prepared.
5. What is the role of alcohol in the saponification reaction?
6. Calculate the yield of the saponification reaction.
7. What is the purpose of the relargage (salting out)?
8. Explain the hydrophilic and hydrophobic parts of the soap.
9. What is the role of reflux heating in this synthesis?
10. Provide a conclusion.