Perform the hydrolysis of esters to form soaps (of varying composition) and glycerol.

Micro-Project Report

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UNDER THE SUPERVISION AND GUIDANCE OF

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ABSTRACT

This study investigates the complete ester reaction cycle, encompassing both esterification and alkaline hydrolysis to analyze the formation and decomposition of esters ranging from simple acetate esters to complex triglycerides. The experimental process includes the synthesis of esters by reacting alcohols (methanol, ethanol, butanol) with acetic acid, followed by base-catalyzed hydrolysis using sodium hydroxide (NaOH). Additionally, natural vegetable oils such as coconut, sunflower, and olive oils were used as triglyceride sources to explore the impact of fatty acid composition on the physical properties of the resulting soap and glycerol.The hydrolysis reactions proceed through nucleophilic attack of the hydroxide ion on the carbonyl carbon of the ester, breaking the ester bond and producing an alcohol and a carboxylate salt. In the case of triglycerides, the process yields glycerol and soap (sodium or potassium salts of fatty acids). This experiment demonstrates how ester chain length, degree of saturation, and feedstock type affect the characteristics of the final soap product — including lathering ability, texture, moisturizing effect, pH. Additionally, the recovery of glycerol (methanol, ethanol, butanol) was carried out and characterized. These recovered by-products contribute to the green chemistry aspect of the process, promoting resource efficiency and biodegradability. The study integrates core organic chemistry principles with practical, sustainable chemical engineering applications, reinforcing the educational and industrial relevance of ester chemistry.

Keywords: Ester, Esterification, Alkaline Hydrolysis, Saponification, Glycerol, Soap Production, Triglycerides, Carbonyl Carbon, Nucleophilic Attack, Alcohol Recovery, Fatty Acid Composition, Green Chemistry, Biodegradable Surfactants, Sustainable Chemical Engineering

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Table of Contents

LIST OF FIGURES

LIST OF TABLES

Chapter 1: Introduction

Soaps and detergents are essential to personal and public health. They safely remove

germs, soils and other contaminants and help us to stay healthy and make our surroundings more pleasant. Soaps are made from fats and oils or their fatty acids. Fatty acids are merely carboxylic acids consisting of a long hydrocarbon chain at one end and a carboxyl group (-COOH) at the other end. They are generally represented as RCOOH. They are an important component of plants, animals and other microorganisms. They are found in various parts of the body, such as cell membranes, the nervous system and as lung surfactant. Soaps are sodium or potassium salts of long chain fatty acids. When triglycerides in fat/oil react with aqueous NaOH or KOH, they are converted into soap and glycerol. This is called alkaline hydrolysis of esters. Since this reaction leads to the formation of soap, it is called the

Saponification process. Hydrolysis is a reaction with water. That is exactly what happens when esters are hydrolysed by water or by dilute acids such as dilute hydrochloric acid. Esters are neutral compounds, unlike the acids from which they are formed. In typical reactions, the alkoxy (OR′) group of an ester is replaced by another group. One such reaction is hydrolysis, literally "splitting with water." The hydrolysis of esters is catalysed by either an acid or a base. The alkaline hydrolysis of esters actually involves reaction with hydroxide ions, but the overall result is so similar that it is lumped together with the other two. The soap molecule has two parts: a polar group (-COO-Na+) and a non-polar group (R- hydrocarbon part). The polar group is called the head and the non-polar group is called the tail. Thus, the soap molecule has a polar head and a non-polar hydrocarbon tail. The polar head is hydrophilic in nature (water loving) and the non-polar tail is hydrophobic (water repelling) in nature.. [1]

A typical example of this reaction is the formation of ethyl acetate.:

Alcohol+ Acetic Acid→ Ester (Acetate)+Water

This reaction is reversible. Esters are known as alkaline ester hydrolysis by using strong foundations such as sodium hydroxide (NaOH) and potassium hydroxide (KOH). If this hydrolysis is carried out with complex esters such as triglycerides (comprised of glycerine and three fatty acids), this leads to the formation of soap (a sodium or potassium salt made of fatty acids) and glycerine.:

Triglyceride+3NaOH→Glycerol+3RCOONa (Soap)

This reaction forms the basis of modern soap production. The properties of the soap produced depend heavily on the type of triglyceride (oil or fat) used as the starting material, such as hardness, racism, and moisturizing properties. For example, coconut oil tends to produce solid bars with plenty of foam, while olive oil leads to soft soaps with better moisture quality [2].

In addition to soaps, glycerine is a valuable ester hydrolysis and has many uses in industries such as pharmaceuticals, cosmetics, and food processing. Advanced techniques such as ultrasound therapy and phase transition catalysts were used to optimize both soap quality and glycerine yield .

Environmental issues and negative effects are increasing with synthetic surfactants, so environmentally friendly soaps from natural plant-based oils are once again in demand. The type of oil used in soap production has been shown to have a major impact on its quality, including moisture quality, cleaning ability, and consistency. For example, olive oil produces softer soaps with wet properties, while coconut oil often produces solid bars with rich foam.

Glycerine recovery is also very industrially important. Its extraction and cleaning can improve the financial sustainability of manual and small soap makers. Methods such as phase transition catalysts and ultrasound therapy have been investigated to increase glycerine production and improve reaction efficiency. [3]

CHAPTER 2: LITERATURE SURVEY

The alkaline hydrolysis technique of esters, specifically triglycerides, remains a essential technique in soap production in commercial and laboratory environments. even though this technique has historic origins, it has gained renewed scientific hobby inside the light of new advances in green chemistry, response performance and innovation of catalysts. upgrades in response speeds, products of the products and the overall sustainability of glycerol and the ensuing soap have been found. further, this approach has multiplied with the advent of non - traditional uncooked substances and ecological method designs

2.1 Mechanism and Basic Chemistry of Ester Hydrolysis

Ester hydrolysis refers to the process in which an ester is decomposed in the presence of a bas e or acidity. In soap production, the hydrolysis of the alkali is applied to the triglycerides. In t riglycerides, the ester is made up of glycerin bound to three fatty acid chains. This reaction re sults in the corresponding sodium or potassium salts of glycerin and fatty acids (i.e. soap).

The general reaction is:

$$\textbf{Triglyceride+3NaOH} \rightarrow \textbf{Glycerol+3RCOONa (Soap)}$$

Mechanically, the hydroxysion nucleophile attacks the carbonyl carbon of the ester group, forming a tetrahedral intermediate which breaks down to release glycerin and soap components. Variables such as fatty acid structure, solvent polarity, temperature, and mixing efficiency strongly influence the yield and efficiency of this reaction.

To better understand this process, it is helpful to consider the reverse reaction: esterification. For example, when an alcohol reacts with acetic acid, an ester (acetic acid) and water are formed.

$$\textbf{Alcohol Acetic Acid} \rightarrow \textbf{Ester (Acetate)+}H_2O$$

Through alkaline hydrolysis, this ester can be broken down again, a principle that scales up in soap-making by using triglycerides instead of simple esters.[4]

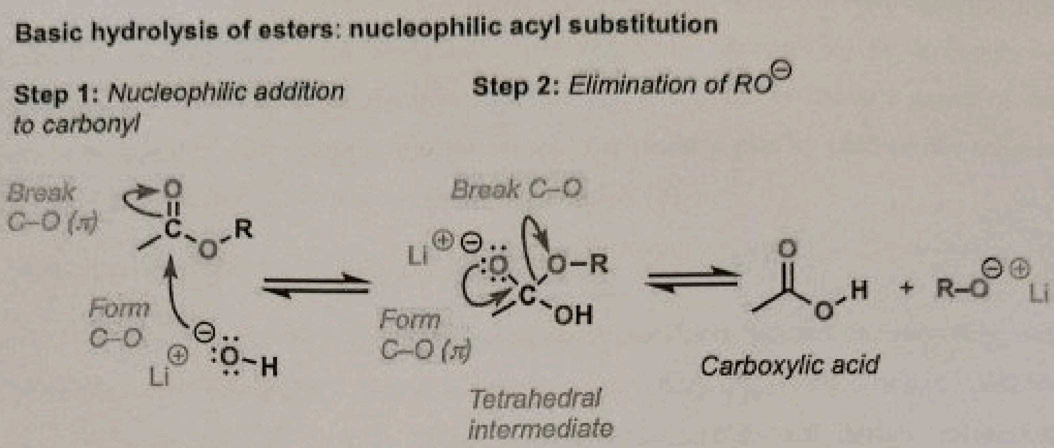

Fig.1 Basic hydrolysis of ester: nucleophilic acyl substitution

Source: Organic Chemistry

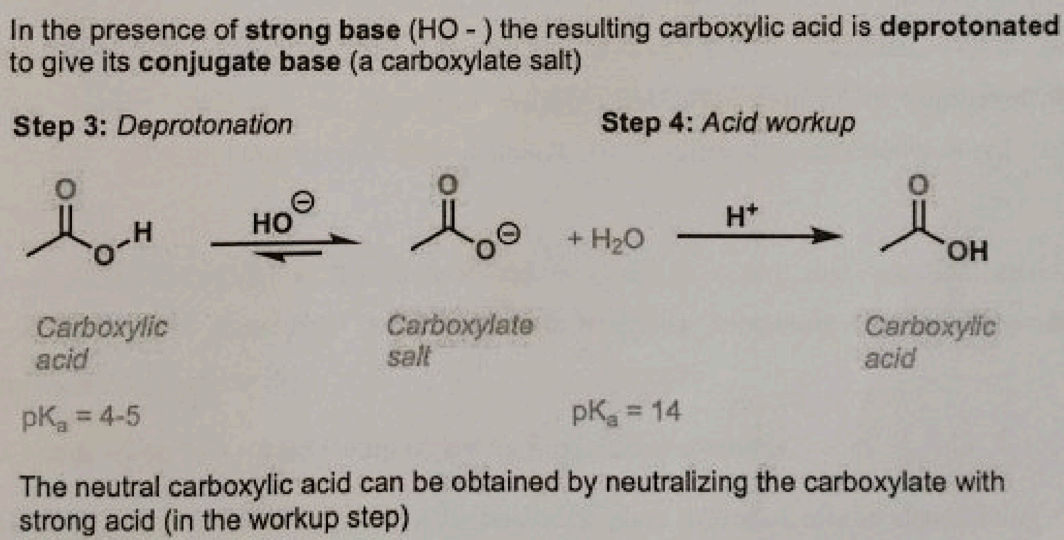

Fig.2 Deprotonated to give conjugate base

Source:Organic Chemistry

2.2 Reaction Kinetics and Optimization Parameters

The hydrolysis of mixed fat mixes consisting of olein and stearin at a 3:1 ratio using sodium hydroxide was investigated in detail using a motility model. Variables such as temperature

(100°C–150°C), stirring speed (400–1100 rpm), and NAOH concentration were adjusted, and effects on reaction rate were observed. Increased water availability of hydroxysion accelerated the hydrolytic factor reaction. Interestingly, due to the exothermic nature of the reaction, excessively high temperatures can reduce the reaction rate by pushing the balance backwards. Motion data is suitable for theoretical models [5].

2.3 Alternative Feedstocks and Non-Traditional Oils

To minimize competition with food supplies, recent studies have focused on renewable, non-edible oil sources. One notable example is Jatropha curcas seed oil, which is rich in lipids and grows widely in tropical regions. Using a D-optimal experimental design, researchers examined how variables such as temperature, reaction time, and KOH concentration influenced hydrolysis efficiency. The maximum yield of soap and glycerol was obtained under optimal conditions: 1.75 M KOH at 65°C for 2 hours [8].

In another case, alkaline extracts derived from wood ash were utilized to hydrolyze oil from Treculia africana (African breadfruit). The reaction followed first-order kinetics, with the rate being directly dependent on the alkali concentration. This experiment confirmed the feasibility of using locally available materials for small-scale, community-based soap production.

These efforts contribute to the advancement of green chemistry principles and circular economy models, particularly in resource-limited regions, supporting broader sustainable development objectives [9].

2.4 Influence of Fatty Acid Composition on Soap Characteristics

The fatty acid composition of the ester feedstock plays a crucial role in determining the physical and functional properties of the resulting soap, such as hardness, foaming capability, solubility, and moisturizing effect. For example, coconut oil, rich in saturated fatty acids like lauric and myristic acids, produces hard soaps with excellent foaming characteristics. In contrast, olive oil, which contains a high percentage of unsaturated fatty acids such as oleic acid, yields softer, more moisturizing soaps.

Castor oil, known for its high ricinoleic acid content, imparts unique emollient and conditioning properties, making it ideal for skincare and cosmetic formulations [10].

By adjusting the fatty acid profile of the oil input, soap manufacturers can tailor formulations to meet specific consumer preferences—from mild facial cleansers to heavy-duty cleaning bars.

2.5 Glycerol: Recovery and Industrial Utility

Glycerol, a key by-product of ester hydrolysis, has numerous industrial applications. While crude glycerol often contains impurities such as residual oil, water, and soap, it can be refined using methods like phase separation, distillation, and neutralization. To achieve high-purity glycerol, further purification steps such as activated charcoal treatment, repeated washing, and drying may be applied.

Due to its broad usage in industries ranging from biofuel production to pharmaceuticals and cosmetics, the recovery of glycerol significantly enhances the overall economic and environmental value of the ester hydrolysis process [11].

2.6 Industrial and Environmental Considerations

Because of its low cost and straightforward requirements, alkaline hydrolysis is still the method of choice in industry. However, research on greener alternatives has been prompted by increased concerns about raw material sustainability, waste management, and energy use. Enzymatic hydrolysis employing lipases is one such method that has advantages such as improved selectivity, reduced environmental effect, and softer operating conditions. Used cooking oil has also been investigated as a potential feedstock, which promotes resource circularity and waste reduction. By lowering carbon emissions and dependence on virgin oils, this approach helps achieve environmental goals [12].

2.7 Gaps in Literature and Future Directions

There are still gaps in the comparative study of feedstocks under consistent experimental circumstances, despite a great deal of research. Direct comparisons are challenging because the majority of research focuses on individual oils or catalysts. Furthermore, not much research has been done on the long-term performance of soaps made from alternative feedstocks in terms of consumer acceptance, shelf life, and biodegradability.

Creating generalized kinetic models that can be applied to various feedstocks is one of the top research priorities for the future.

- Researching how trace elements, such as sterols, tocopherols, and pigments, affect the quality of products.
- To improve sustainability, solar energy and other renewables should be integrated.
- Assessing the viability of several glycerol recovery techniques from an economic and environmental standpoint [13]

CHAPTER 3: METHODOLOGY

3.0 Objective

This experiment's goal is to make soap with different compositions and glycerol by performing the alkaline hydrolysis of esters, specifically triglycerides and simple esters. The goal of the study is to show how ester bonds break down in simple situations and how the alcohol's makeup, which is utilized to generate ester, affects the result of ester hydrolysis.

3.1 Materials Used

The following materials and chemicals were used in the ester hydrolysis experiment:

Oils (Triglyceride Sources): Coconut oil – high in lauric acid, yields hard soap with good foaming

Alcohols for Ester Formation:

- Methanol
- Ethanol
- Butanol

Acid: Acetic acid – used for esterification with different alcohols

Alkali:

- Sodium hydroxide (NaOH) pellets – used as the hydrolyzing agent

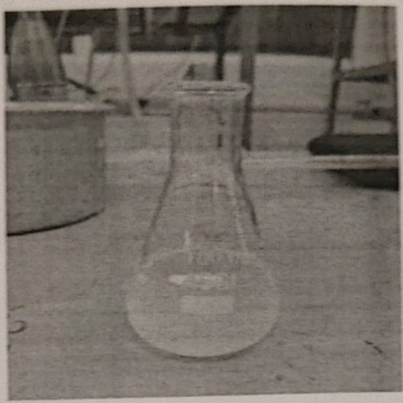

Fig 3: Sodium hydroxide

- Potassium hydroxide (KOH) – optionally used to produce liquid soap in selected trials

Solvent:

- Distilled water – for dissolving NaOH and sample dilution

Lab Equipment:

- Beakers (100 mL, 250 mL): Used to hold and mix liquids.
- Conical flasks: Used for mixing and storing the reaction mixture.
- Glass rods: Used for manually stirring liquids.
- Hot plate with stirrer: Used to heat and mix solutions evenly.
- Digital weighing balance: Used to accurately measure solid chemicals.
- Thermometer: Used to monitor the temperature during the reaction.
- Measuring cylinders: Used for measuring liquid volumes precisely.
- Batch reactor: Used to perform the ester hydrolysis reaction.

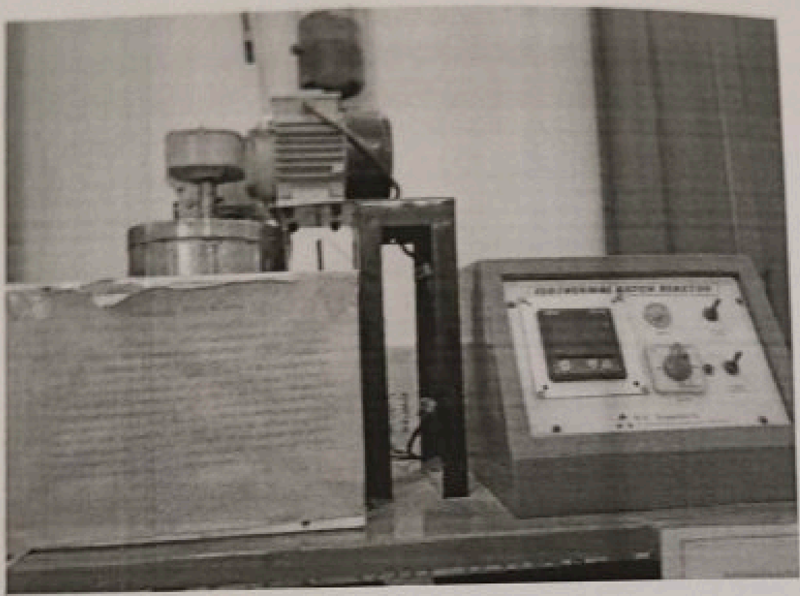

Fig.4 : Batch reactor

- pH paper or pH meter: Used to check the alkalinity of the final soap.
- Filter paper and funnel: Used to separate solid soap from the liquid mixture.
- Ice bath: Used to quickly cool down reactants if needed.
- Soap mold trays: Used to shape and set the final soap product.

3.2 Experimental Procedure: Alkaline Hydrolysis of Esters (Cold Process)

This methodology consists of two parts:

A. Synthesis of Esters with Varying Alcohols:

1. 1. Methyl acetate, ethyl acetate, and butyl acetate were the products of distinct reactions between acetate and methanol, ethanol, and butanol in an acidic environment at a molar ratio of 1:1.
2. To finish the esterification, the mixture was slowly heated and refluxed for an hour.
3. Following separation and purification, the esters were prepared for hydrolysis.

B. Alkaline Hydrolysis of Triglycerides and Esters:

This procedure describes the cold-process method for hydrolyzing triglycerides to form soap and glycerol using 100% coconut oil.

1. **Safety and Preparation:**

   A ventilated workspace was set up, and PPE was used throughout:
   - Protective eyewear
   - Rubber or nitrile gloves
   - Full-sleeved lab coat

   Because of its corrosive properties, NaOH was handled carefully. A digital balance with 0.1 g precision was used to weigh each reagent. NaOH was prepared exclusively in non-reactive containers made of heat-resistant plastic or stainless steel.

2. **Preparation of Alkaline Solution:**

   62.4 g of distilled water was carefully mixed with 33 g of NaOH pellets (never the other way around), and the mixture was stirred until the pellets were completely dissolved. The solution was chilled to 35–40°C prior to use because the exothermic process increased the temperature.

3. **Preparation of Triglyceride (Oil) Phase:**

   To get the alkaline solution temperature, 240 g of coconut oil was heated gradually until completely liquefied and then cooled to 35–40°C.

4. **Combining Ester and Alkaline Phases:**

   With constant stirring, the melted coconut oil was gradually mixed with the NaOH solution. Short bursts of an immersion blender were utilized to encourage mixing. The liquid attained "trace"—a discernible thickening signifying the advancement of ester hydrolysis—after the process was prolonged.

   Separately, stoichiometric amounts of the pure esters (butyl, ethyl, and methyl acetate) were added to a NaOH solution to hydrolyze them.

   Gentle heating was applied to each reaction until phase separation or completion was noted.

Keep mixing for a minute until you reach trace. Trace is when the oil and water combine, it makes a thick emulsion. Don't use the blender for too long because you don't want to reach over trace (when the emulsion is too thick to pour).

5. **Addition of Optional Fragrance:**

   7 g of rose essential oil was added during trace. It was stirred gently to ensure even distribution.

6. **Molding and Setting:**

   Clean silicone molds were filled with the hydrolyzed mixture. Air bubbles were eliminated with gentle tapping. To allow the molds to firm, they were covered and left alone for 24 to 48 hours.

7. **Curing:**

   Following demolding, soap bars were allowed to cure in a ventilated area for three to six weeks. This made it possible for the pH to stabilize (usually between 8 and 10), moisture to evaporate, and hardness to form.

8. **pH Testing and Product Evaluation:**

   To check the PH of the soap, wet one side of the soap, then place a PH strip on the damp side of the soap. to confirm skin safety. Products were observed for texture, hardness, and fragrance retention [14]

CHAPTER 4: RESULTS

4.1 Visual Observations:

- The reaction mixture thickened after 5–7 minutes after adding coconut oil and NaOH, showing the breaking of ester bonds and the creation of soap.
- For methyl, ethyl, and butyl esters, phase separation was observed post-hydrolysis, confirming successful breakdown of each ester into its alcohol and acid salt.
- All soap mixtures displayed a creamy white appearance and homogeneous texture.
- No phase instability or layer separation occurred in any of the batches.

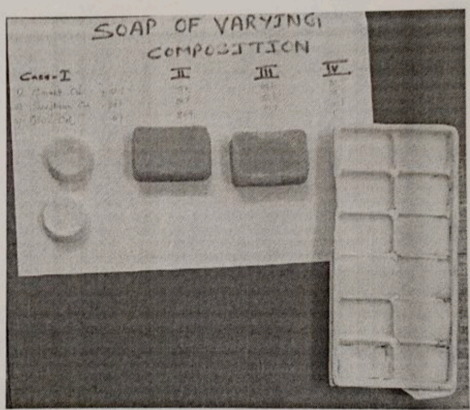

Fig.3 Soap of varying composition

4.2 pH Testing:

• The hydrolysis of methyl acetate, ethyl acetate, and butyl acetate also produced aqueous products with mildly alkaline pH (8–9), suggesting good ester cleavage

• All cured soap bars had pH values between 8.5 and 9.5, indicating mild alkalinity appropriate for skin usage.

4.3 Soap Bar Characteristics (Coconut Oil):

Property	Observation
Texture	Smooth and hard
Color	White, opaque
Lathering ability	High
Fragrance retention	Mild rose scent preserved
Curing time	4 weeks for optimal hardness
Solubility in water	High; quick dissolution in warm water
Moisturizing feel	Moderate
Skin compatibility	No irritation, gentle on skin

4.4 Glycerol Recovery:

- A viscous, colorless liquid layer identified as **glycerol** was collected during soap separation from coconut oil.
- In hydrolysis of simple esters:
    - **Methanol** was recovered from methyl acetate.
    - **Ethanol** from ethyl acetate.
    - **Butanol** from butyl acetate.

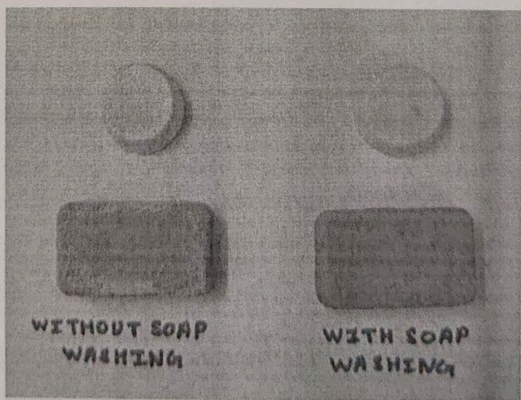

Fig.4 Soap washing

4.5 Table: Experimental Observations and Predicted Outcome

Sample Type	Feedstock	Visual Observation	Physical Property Outcome	pH Range	Product Yield	Notes / Prediction
Methyl acetate	Methanol + Acetic acid	Clear phase separation	Low viscosity, fast hydrolysis	8.5–9.0	Moderate	Methanol recovered; high solubility
Ethyl acetate	Ethanol + Acetic acid	Slight odor, clear separation	Smooth product; slightly slower hydrolysis	8.5–9.0	Moderate	Ethanol recovered; stable separation
Butyl acetate	Butanol + Acetic acid	Slow separation, oily texture	Viscous layer, slow hydrolysis	8.0–8.5	Moderate	Longer chain causes slower breakdown
Triglyceride Soap	Coconut oil	Thick opaque paste (trace) → solid bar	Hard bar, high foaming, moderate moisturizing	8.5–9.5	High	Glycerol recovered; soap cured in 4 weeks
Triglyceride Sssoap	Sunflower oil	Slower trace, softer mixture	Soft bar, better moisturizing, low foaming	8.0–9.0	Moderate	More unsaturated fats = softer soap
Triglyceride Soap	Olive oil	Very soft trace, creamier appearance	Gentle soap, high moisturizing, soft texture	8.0–8.5	Moderate	Excellent skin feel; longer cure time

This data confirms successful alkaline hydrolysis of both simple esters and triglycerides, producing soap and glycerol or corresponding alcohols as expected.[15]

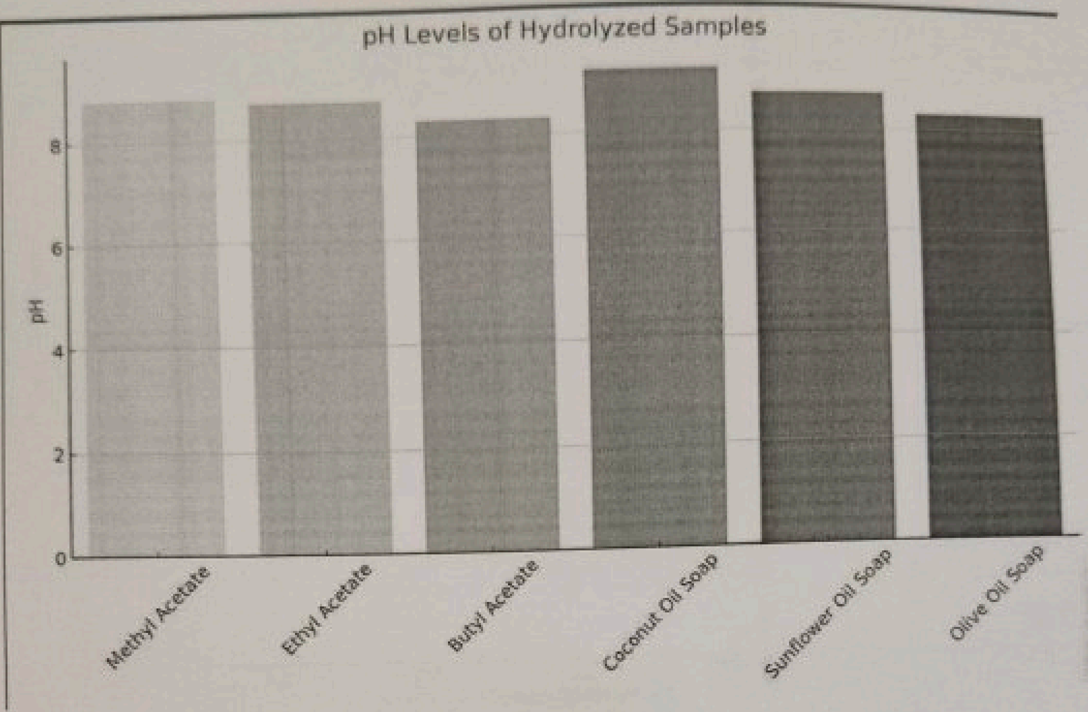

Fig.5 Bar Chart: pH levels across all ester and soap samples

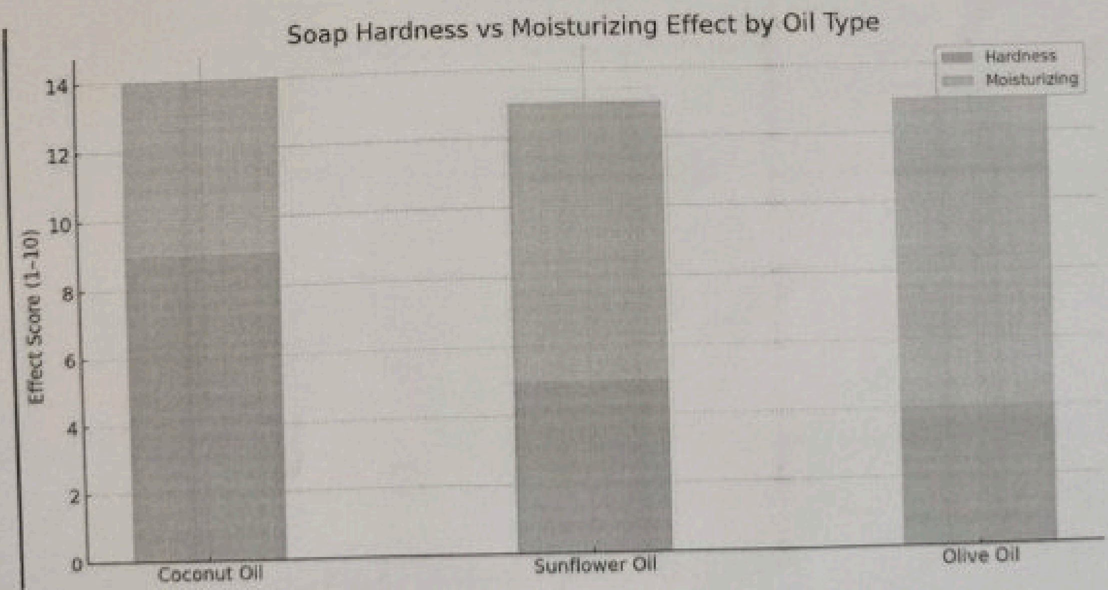

Fig.6 Stacked Bar Chart: Soap hardness vs moisturizing effect for coconut, sunflower, and olive oils.

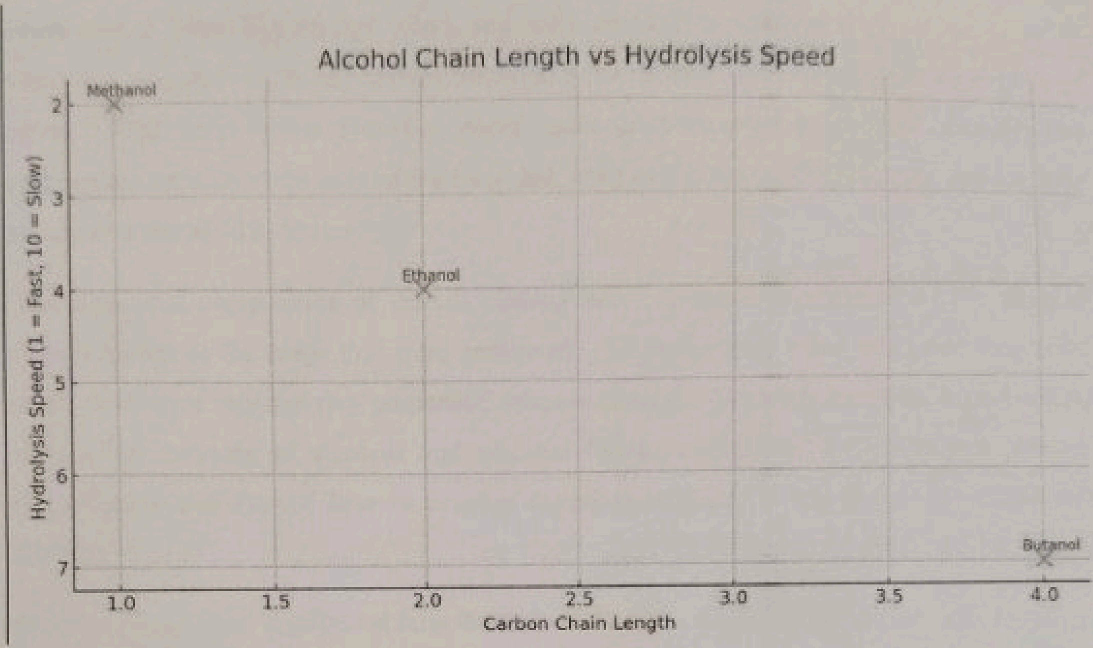

Fig.7 Scatter Plot: Shows how alcohol chain length affects hydrolysis speed (longer chains = slower hydrolysis).

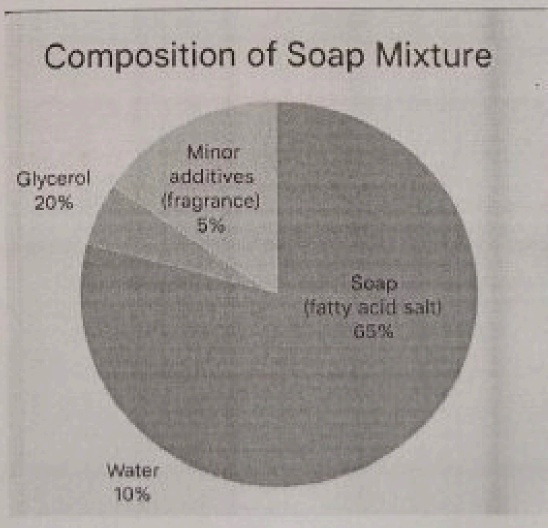

Fig.8 Composition of soap mixture.

CHAPTER 5 : CONCLUSION

From simple esters like methyl, ethyl, and butyl acetates to complex triglycerides found in vegetable oils, this study effectively illustrated the production and alkaline hydrolysis of esters. With ester bonds breaking under basic circumstances to produce alcohols and carboxylate salts, or in the case of triglycerides, soap and glycerol, the reactions validated the anticipated chemical behavior.

The fatty acid composition of the oil utilized had a considerable impact on the physical characteristics of the soaps that were generated. Sunflower and olive oils generated softer soaps with more moisturizing properties, whereas coconut oil produced firm, high-foaming soap. The recovery of alcohols and glycerol further established the hydrolysis process' effectiveness and showed how to develop chemical products in an integrated, sustainable manner.

The experiment also highlighted how the type of alcohol, degree of saturation, and length of the ester chain affect reaction rate and product quality. The method's dependability was confirmed by observations of pH, texture, solubility, and curing behavior that agreed with data provided in the literature.

All things considered, this work demonstrated the potential of green chemistry in creating environmentally friendly, biodegradable surfactants in addition to expanding our knowledge of ester hydrolysis and its economic significance. The study demonstrates how straightforward organic reactions can be used in both commercial and scientific settings to produce high-value, sustainable products.[16]

SOCIAL RELEVANCE

This study addresses critical aspects of public health, environmental sustainability, and rural economic empowerment through the lens of ester hydrolysis and biodegradable soap production. By utilizing non-edible vegetable oils and natural feedstocks, the process reduces dependency on synthetic surfactants, which are often harsh on skin and harmful to aquatic ecosystems.

Moreover, the recovery of glycerol, a versatile by-product, adds economic value and promotes resource efficiency. The methodology supports low-cost, small-scale production, making it highly applicable for local communities and developing regions with limited access to commercial hygiene products.

Encouraging the use of green chemistry in everyday products like soap fosters greater environmental awareness and aligns with sustainable development goals (SDGs), particularly in the areas of clean water and sanitation (SDG 6), responsible consumption and production (SDG 12), and good health and well-being (SDG 3).[17]

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