

CHAPTER I

INTRODUCTION

1.1 Background

Fats and oils are triglycerides, which are triesters of glycerol. The chemical properties of oils and fats are determined by their solubility. At room temperature, fats are solid or semi-solid triglycerides, while oils are liquid triglycerides or clear liquids. After carbohydrates and proteins, fats and oils are the three main food groups (Ogori, 2020). Most triglycerides in animals are fats, commonly referred to as animal fats. Meanwhile, triglycerides in plants tend to be oil and are referred to as vegetable oil.

The castor plant (*Ricinus communis*) is a source of renewable oil and is classified as a non-edible oil, meaning it does not compete with human consumption needs such as palm oil, corn oil, and others. The main product of the castor plant is its seeds. When dried, castor seeds produce castor oil. The hydrolysis of castor oil into fatty acids and glycerol is carried out by heating a mixture of castor oil and a small amount of sulphuric acid. The use of sulphuric acid, which is a strong acid, is required as a catalyst because the hydrolysis reaction proceeds very slowly. The fatty acids obtained from the hydrolysis of an oil or fat generally have long, unbranched carbon chains.

The direct use of castor oil is generally limited to roofing tiles, medicine, brake fluid, and lubricant industries. The hydrolysis products of castor oil are commonly used in the cosmetics, pharmaceutical, oleochemical, textile, and paper industries, as well as solvents in the food and beverage industries (Panjaitan *et al.*, 2024).

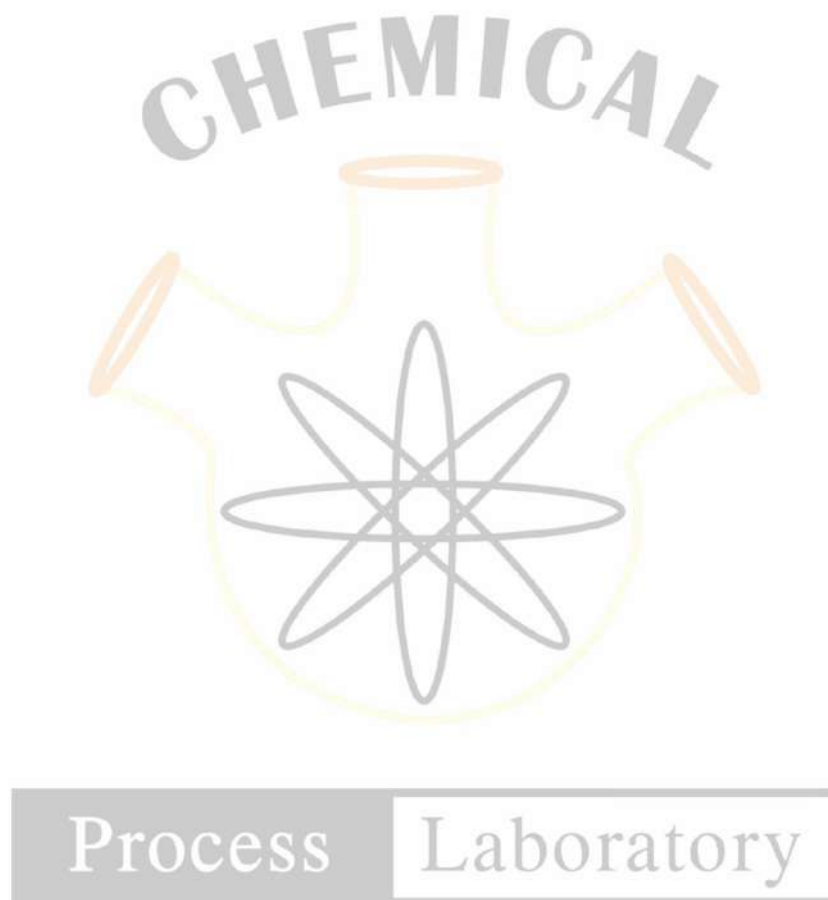
1.2 Practice Objectives

After doing this experiment, practitioners are able to explain the following things:

1. The effect of variables on the conversion of castor oil hydrolysis (X_A).
2. The effect of variables on the reaction rate constant of castor oil hydrolysis (k).
3. The effect of variables on the equilibrium direction of castor oil hydrolysis (K).

1.3 Practice Benefits

1. Understand the effect of variables on the conversion of castor oil hydrolysis (X_A).
2. Understand the effect of variables on the reaction rate constant of castor oil hydrolysis (k).
3. Understand the effect of variables on the equilibrium direction of castor oil hydrolysis (K).



CHAPTER II

LITERATURE REVIEW

2.1 Castor Oil

Castor (*Ricinus communis*) is an annual herbaceous plant commonly found in tropical and subtropical regions. Castor oil is a vegetable oil obtained by pressing the seeds of this plant (Nitbani *et al.*, 2022). The direct use of castor oil is generally limited to roofing tiles, pharmaceutical, brake fluid, and lubricant industries.

The physical properties of castor oil are a colourless or pale-yellow liquid, with a fatty odour, a slightly pungent taste, high viscosity, and a high acid number, which is indicated by damaged seeds and poor extraction methods. The chemical properties of castor oil are that it contains 46%-53% oil. Castor oil contains 80% glycerides, asinolate acid, isoeresinolate stearate, dihydroxy stearate, and palmitate. Castor oil also contains 20% protein, 0,2% toxic pyridine alkaloids, ricin, and the enzyme lipase. Castor oil contains the toxin substance ricin.

Castor oil extraction is carried out mechanically, using hydraulics at room temperature. Dry castor beans are first heated using an autoclave at a temperature of 125°C-145°C for 30 minutes. The extraction process is then carried out at a pressure of 200 kg/cm²-400 kg/cm² for 5 minutes-15 minutes. The extraction yield is crude castor oil, which requires further processing to be used for various industrial purposes (Mamudu *et al.*, 2019).

Table 1. Fatty acid composition of castor oil

Type of fatty acid	Composition (%)
Ricinoleic acid	87,3
Oleic acid	4,69
Linoleic acid	4,92
Stearic acid	1,241
Palminic acid	1,016

(Yeboah *et al.*, 2021)

2.2 Castor Oil Hydrolysis

Hydrolysis is the binding of hydroxyl groups (OH⁻) by a compound. OH⁻ groups can be obtained from water. Hydrolysis can be classified into pure hydrolysis, acid catalyzed hydrolysis, base catalyzed hydrolysis, and

enzyme-catalyzed hydrolysis. Based on the reaction phase, hydrolysis is grouped into liquid phase hydrolysis and vapor phase hydrolysis.

The hydrolysis of castor oil into fatty acids and glycerol is carried out by heating a mixture of castor oil and a small amount of strong acid in a three-necked flask. A strong acid is required as a catalyst because the hydrolysis reaction proceeds very slowly. A catalyst is not required if hydrolysis is carried out at very high pressure, specifically 700 psia and 485°F (Groggins, 1985), and the conversion achieved is > 90%. In the hydrolysis process, water breaks down the alkyl groups in the triglycerides of the oil into fatty acids and glycerol. Heating is carried out until the desired temperature is reached before hot water is added. Samples are taken at regular intervals to analyze the free fatty acid content. The rate of hydrolysis is primarily determined by the reaction rate between water and triglycerides in the oil phase. Excessive use of water allows the oil phase to remain saturated with water, resulting in a pseudo-first-order hydrolysis reaction with respect to glyceride concentration. Generally, the fatty acids produced by hydrolysis are straight-chain and have an even number of carbon atoms. If there are double bonds, they are typically in the cis (or Z) configuration and are not conjugated (Hart, 1983).

Reactions with water can occur in both the liquid and oil phases (Lewkowitsch, 1903). However, according to Lascaray (1949), reactions in the oil phase are more dominant, so that the kinetics of the reaction are determined by the rate of diffusion of water into the oil phase and the reaction between water and oil in the oil phase, which can be expressed mathematically as follows.

- a. The rate of diffusion of water into the oil phase

$$-r_A = k_{1A}(C_A^* - C_{A1}) \text{ mgreq/g oil/min} \quad (1)$$

With: C_A^* states for concentration of water in the oil phase is equilibrium to the concentration of water in the aqueous phase or $C_A^* = k \cdot C_{A2}$, C_{A1} states for concentration of water in the oil phase (mgreq/g oil), C_{A2} states for concentration of water in the liquid phase, and k_{1A} states for rate of diffusion constant of water to the oil phase (min).

- b. Rate of reaction in the oil phase

$$r_A = r_B = k_r \cdot C_{A1} \cdot C_B \quad (2)$$

With: C_{A1} states for concentration of water in the oil phase and C_B states for concentration of oil/triglycerides.

- c. Mass balance of water in the oil phase

To find the step that controls the reaction kinetics, mass balance of is put out as follows:

$$\frac{dC_{A1}}{dt} = k_{1A}(C_A^* - C_{A1}) - k_r \cdot C_{A1} \cdot C_B \quad (3)$$

Assumption: with stirring, the mass transfer rate in the equation (3) $[(k_{1A}(C_A^* - C_{A1}))]$ is assumed much greater compared to the chemical reaction rate $[k_r \cdot C_{A1} \cdot C_B]$ so it is considered that only the rate of chemical reaction determines the rate of the overall reaction.

- d. Mass balance of oil in the oil phase

$$\frac{dC_{A1}}{dt} = -k_r \cdot C_{A1} \cdot C_B \quad (4)$$

With the assumption that the reaction is first order, then one of the reactants is made excess. If the amount of water is excessive and the mass transfer of water to the oil phase is very fast, then the oil phase is considered to be always saturated with water, then $C_{A1} = C_A'$ which is constant at a certain temperature, $k_r \cdot C_{A1} = k'$ so $-r_B = k' \cdot C_B$.

$$\begin{aligned} \frac{dC_{A1}}{dt} &= \frac{dC_B}{dt} = -k' \cdot C_B \\ \int_{C_{B0}}^{C_B} \frac{1}{C_B} dC_B &= -k' \int_0^t dt \\ \ln \frac{C_B}{C_{B0}} &= -k't \end{aligned} \quad (5)$$

With: C_{B0} states the initial amount of triglycerides (mgreq/g oil) and C_B states the amount of triglycerides at a certain time is C_{B0} minus the amount of free fatty acids formed (mgreq/g oil).

If $X = \frac{\text{the amount of free fatty acids formed}}{\text{the initial amount of triglycerides}}$, then

$$X = \frac{C_{B0} - C_B}{C_{B0}}$$

$$X = 1 - \frac{C_B}{C_{B0}}$$

$$\frac{C_B}{C_{B0}} = 1 - X \quad (6)$$

$$\ln(1 - X) = -k't \quad (7)$$

With: k states for the first order of rate constant (s^{-1}) and t states for the time of the reaction (s).

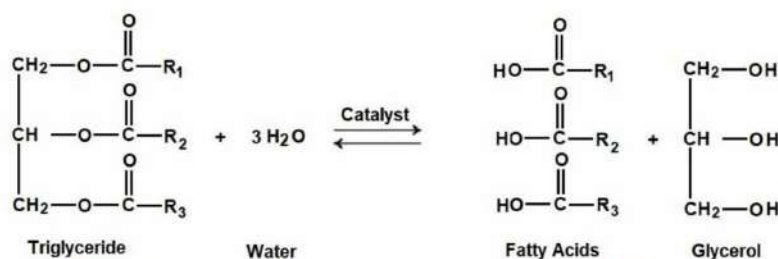
The value of chemical reaction rate constant as a function of temperature can be expressed by the Arrhenius equation:

$$k = Ae^{\frac{E_a}{RT}} \quad (8)$$

With: k states for the first order of rate constant (s^{-1}), T states for temperature (K), R states for gas constant, and E_a states for activation energy.

2.3 Mechanism of Castor Oil Hydrolysis

Hydrolysis is a process of breaking down compounds using water. Castor oil is triglycerides of fat, which when hydrolyzed by water will break down to free fatty acids and glycerol. The hydrolysis formula can be seen in Picture 1. (Gunawan *et al.*, 2019).



Picture 1. Triglyceride hydrolysis reaction

The mechanism of castor oil hydrolysis with a catalyst follows the breakdown of the ester. Free fatty acids are removed from the glyceride molecule, so the breakdown of fats does not run completely. The cleavage occurs between the surface of the oil and fat, which is a homogeneous reaction through the oxidation of water dissolved in the oil phase (Fessenden, 1999).

2.4 Factors Affecting Castor Oil Hydrolysis

1. Time of The Reaction

The longer the reaction occurred, the more opportunities for substances to react so that the conversion is greater. When the reaction equilibrium is reached, increasing the reaction time will not increase the conversion.

2. Temperature

Increase in temperature will increase the value of the reaction rate constant. The higher the temperature will increase the solubility of water in the oil phase, so that more triglycerides react. According to Rahayu (1999), the relation between the reaction rate constant and temperature can be expressed by the equation:

$$k = 1,2515 \cdot 10^8 e^{\frac{-8022}{T}} = 1,2515 e^{\frac{-15939}{RT}} \quad (9)$$

With: k states for the first order of rate constant (s^{-1}), T states for absolute temperature (K), R states for gas constant are 1,987 cal/gmol K.

3. Catalyst

Catalyst is a substance added to a reaction to increase the rate of the reaction. The purpose of a catalyst is to increase the speed of the reaction (accelerate the reaction) by reducing the activation energy of a reaction. The lower the energy activation, the faster the reaction can take place at the same temperature. Catalysts that can be used are enzymes, acids, bases, salts, and ion exchangers. The catalyst chosen in this experiment is acid catalyst. The more acid catalyst is added, the greater the conversion as well as the reaction rate constant. When the catalyst is added, the more triglyceride molecules are activated.

According to Rahayu (1999), the relation between reaction rate constant (K_c) and acid concentration (c) mg mole of H_2SO_4 / gr oil can be expressed by the equation:

$$K_c = 0,14525 c^{13} \quad (10)$$

with c states for mg mole of H_2SO_4 / gr oil

Catalysts can lower activation energy (E_a) by changing the mechanism of the process. Catalysts lower E_a by providing alternative reaction pathways (intermediates) that involve lower energy barriers, so that the energy required to convert reactants into products is lower (Isahak & Al-Amiery, 2024).

4. Stirring

In order for substances to collide with each other properly, it is necessary to do stirring. For batch processes, this can be achieved with the aid of a stirrer. If the process is continuous, the stirring is done by adjusting the flow in the reactor so that agitation occurs. The reaction can run well if the solution is mixed properly. Stirring can reduce the mass transfer resistance and speed up the diffusion of the reactants (Fogler, 1999).

5. Ratio of Reactants

If one of the reactants is produced in excess, the equilibrium will shift towards the product (right). Conversely, if the product is removed, the reaction will shift to the right, thereby increasing the collision frequency and the reaction rate constant (Quílez, 2021).

2.5 The Effect of Surfactant

In the hydrolysis of castor oil, the surfactant used is an emulsifier in the form of soap. Surfactants are amphiphilic molecules that have two different groups: a hydrophilic group (water-loving) and a lipophilic group (oil-loving), enabling them to unite a mixture of oil and water. Typically, the nonpolar (lipophilic) part consists of a long alkyl chain, while the polar (hydrophilic) part contains a hydroxyl group (Khalfallah, 2024).



Picture 2. Surfactant structure formula

Within a surfactant molecule, one group must be dominant in number. If the polar group is more dominant, the surfactant molecule will be absorbed more strongly into water than into oil. This causes the surfactant to be absorbed more on the surface of the water, resulting in a decrease in surface tension between the water and oil. As a result, the system becomes more stable, with one phase (water) forming a continuous phase, while the other phase (oil) is dispersed in the form of small droplets. Conversely, if the nonpolar group is more dominant, the surfactant molecule will be more strongly absorbed by oil than by water (Indirasvari, 2018).

2.6 The Usage of Ethanol

Ethanol or ethyl alcohol, which is considered the main ingredient in alcoholic beverages, is a non-volatile, colourless, and flammable liquid with the chemical formula C_2H_5OH . Ethanol has a sharp taste with a pH of 7,33 and a molecular weight of 46,07 as a polar solvent for lipid molecules (Yavuz, 2021).

The function of alcohol is to destroy oil containing cells and dissolve the oil. The requirements for a compound to be used as a solvent include being inert, able to dissolve but not react with oil components, and having a low enough boiling point so that the solvent can be easily evaporated without using high temperatures (Cahyono & Suzeri, 2018).

CHAPTER III

PRACTICAL METHODOLOGY

3.1 Experimental Design

3.1.1 Practical Scheme

3.1.2 Variables

- a. Control variables
 1. Volume of mixture :
 2. Volume of emulsifier :
 3. Time interval :
 4. NaOH concentration :
 5. Titration temperature :
 6. $\text{H}_2\text{SO}_4/\text{HCl}$ concentration :
- b. Independent variables
 1. Catalyst concentration
 2. Temperature
 3. Type of catalyst
 4. Mole ratio of castor oil and water

3.2 Materials and Tools Used

3.2.1 Materials

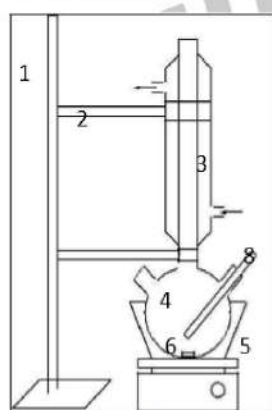
- a. Castor Oil ... mL
- b. Aquadest
Using H_2O from the Reverse Osmosis (RO) process ... mL.
- c. Catalyst
The catalyst is H_2SO_4 acid catalyst with a purity of 98% or HCl ... N ... mL.
- d. NaOH
Using technical grade NaOH in the form of crystals and white in color, produced by PT. BRATACO CHEMIKA ... gram.
- e. Alcohol
Using ethanol atau methanol dengan with a purity of ...% ... mL.
- f. Surfactant
Using Sunlight produced by PT. UNILEVER Tbk. ... mL.
- g. Indicator of titration using 3 drops of PP.

3.2.2 Tools

- a. Three neck bottle flask

- b. Stative
- c. Clamp
- d. Burette
- e. Heater and magnetic stirrer
- f. Thermometer
- g. Condenser
- h. Waterbath
- i. Erlenmeyer

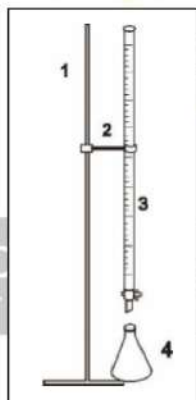
3.3 Picture of The Tools



Description:

- 1. Stative
- 2. Clamp
- 3. Condenser
- 4. Three neck bottle flask
- 5. Waterbath
- 6. Magnetic stirrer
- 7. Heater
- 8. Thermometer

Picture 3. Series of hydrolysis equipment



Description:

- 1. Stative
- 2. Clamp
- 3. Burette
- 4. Erlenmeyer

Picture 4. Series of titration equipment

3.4 Response Test Results

The response taken was the level of free fatty acids formed as a result of castor oil hydrolysis so that the conversion rate, reaction rate constant, and equilibrium constant could be determined.

3.5 Procedures

A. Density Measuring

1. Density of Castor Oil

Weigh the empty picnometer (m_1), pour castor oil into the picnometer with a known volume (V), and weigh it (m_2). Calculate the density of castor oil.

$$\rho = \frac{m_2 - m_1}{V} \quad (11)$$

2. Density of Catalyst

Weigh the empty pycnometer (m_1), pour technical HCl from the laboratory into the pycnometer with a known volume (V), and weigh it (m_2). Calculate the density of the HCl catalyst. Do the same for H_2SO_4 .

B. Analysis of Free Fatty Acid Content in Crude Castor Oil

1. Put ... mL of castor oil into the erlenmeyer.
2. Add ... mL of solvent and heat it while stirring at the temperature of ... °C.
3. Add 3 drops of PP indicator and titrate the solution with NaOH until the color changes to pink.
4. Write down the volume of titrant needed.

C. Castor Oil Hydrolysis

1. Put castor oil into the three neck bottle flask.
2. Put the catalyst into the three neck bottle flask.
3. Flow cooling water during the hydrolysis process.
4. Heat the solution to the temperature of ... °C, then add heated aquadest and emulsifier into the three neck bottle flask.
5. Take sample in an interval of ... minutes to analyze the amount of fatty acids and total acids formed for ... minutes.

D. Determination of Free Fatty Acid Content After Hydrolysis

1. Put ... mL of the hydrolyzed castor oil into the erlenmeyer.
2. Add ... mL of solvent and heat it while stirring at the temperature of ... °C.
3. Add 3 drops of PP indicator and titrate the solution with NaOH until the color changes to pink.
4. Write down the volume of titrant needed.
5. Repeat the hydrolysis steps and analyze the free fatty acid content of the hydrolysis results according to the variables.

3.6 Calculation Method

A. Analysis of Free Fatty Acid Content in Crude Castor Oil

1. Calculate %AL

$$\%AL = \frac{(V \times N) \text{ NaOH} \times \text{MW oil}}{(\rho \times V) \text{ oil} \times 1000} \times 100\% \quad (12)$$

2. Calculate %Glyceride

$$\%Glyceride = 100\% - \%AL = \dots\% \quad (13)$$

3. Calculate Initial Free Fatty Acid (a)

$$a = \frac{(V \times N) \text{ NaOH}}{(\rho \times V) \text{ oil}} \quad (14)$$

4. Calculate Initial Glyceride (b)

$$b = \frac{\%Glyceride}{\%AL} \times a \text{ mole} \quad (15)$$

B. Determination of Free Fatty Acid Content After Hydrolysis

1. Calculate Fatty Acid Hydrolysis

$$C_{A\text{hydrolysis}} = \frac{(V \times N) \text{ NaOH} - (V \times N) \text{ catalyst}}{(\rho \times V) \text{ oil}} \quad (16)$$

2. Calculate Free Fatty Acid Formed

$$C_{A\text{formed}} = C_{A\text{hydrolysis}} - C_{A\text{before hydrolysis}} \quad (17)$$

3. Calculate Reactive Glyceride (d)

$$d = \frac{1}{3} \times C_{A\text{formed}} \quad (18)$$

4. Calculate Triglyceride Conversion (X_A)

$$X_A = \frac{\text{Mole of Reactive Glyceride}}{\text{Mole of Initial Glyceride}} \quad (19)$$

Process

Laboratory

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Process

Laboratory

HAZARD IDENTIFICATION AND RISK ANALYSIS
MATERIAL: CASTOR OIL HYDROLYSIS

HAZARD IDENTIFICATION (HI)										
A	Mechanical		D	Environment		E	Chemical		G	Other risks
A1	Manual handling		D1	Noise		E1	Toxicant	√	G1	Compressed gas
A2	Moving parts		D2	Vibration		E2	Irritant	√	G2	Ionising radiation
A3	Rotating parts	√	D3	Lighting		E3	Corrosive	√	G3	UV radiation
A4	Cutting		D4	Humidity		E4	Carcinogenic		G4	Fatigue
B	Biology		D5	Temperature	√	E5	Flammable	√	G5	Cramped space
B1	Bacteria		D6	Dangers of travel		E6	Explosive	√	G6	Crowded
B2	Virus		D7	Slippery surface	√	E7	Cryogenics		G7	Thermometer
B3	Mold		D8	Solid waste		F	Equipment			
C	Electrical		D9	Air quality		F1	Pressure vessel			
C1	High voltage	√	D10	Solitary work		F2	Heating equipment	√		
C2	Static electricity		D11	Splashes/drops/floods	√	F3	Laser			
C3	Cable	√	D12	Pile of powder		F4	Glass vessel			

Process

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RISK ANALYSIS								
IB	Risks (after control measures)				Risk identification	Control measures to minimise risk	First aid measures	
	High	Medium	Low	Minimal				
1. PREPARATION / INITIAL PRACTICAL								
D7. D11				√	When calibrating the pycnometer and preparing reagents, there is a risk of distilled water spilling, causing the surface to become slippery.	Be careful when using distilled water. If distilled water is being poured into an area prone to spills, use a funnel to minimise the risk of slippery surfaces.	If you slip, check the injured area and treat it. If the injury is serious or worsens, take the victim to the nearest hospital or clinic.	
E5. E6			√		When titrating free fatty acids on crude castor oil with alcohol solvents, there is a risk of the alcohol catching fire and exploding.	Ensure that alcohol does not come into direct contact with heat sources/flame sources. Be careful when using alcohol so that it does not spill; you can use a funnel as a tool to help pour the alcohol.	Extinguish the fire with water or place a wet cloth over the source of the fire. If the fire is large enough, use a fire extinguisher to put it out.	
C1. C3			√		When titrating free fatty acids on crude castor oil, the solution is heated with an electric stove. Flaked wires can cause electric shock.	Check before use to ensure that the tool is in good condition.	Unplug the power source to stop the flow of electricity.	

Process

Laboratory

2. MAIN PRACTICAL							
A3				√	A magnetic stirrer rotating too fast inside a three-necked flask can cause the flask to break.	Ensure that the magnetic stirrer scale is not too high or overpowered.	Turn off the magnetic stirrer and stop the experiment.
C1. C3			√		Exposed electric stove cables can cause electric shocks.	Check before use to ensure that the tool is in good condition.	Unplug the power source to stop the flow of electricity.
D5. F2			√		Contact with electric stoves or heated appliances during hydrolysis.	Be careful with hot surfaces when heating and place the equipment in a safer place to avoid contact.	Stop the heating process, cool the burn, and administer pain medication. If the pain persists, take the patient to the nearest clinic or hospital.
E1. E2		√			Reagents such as castor oil, catalysts, and surfactants are toxic and irritating.	Be careful when using/pouring reagents so as not to expose them to body parts, especially the skin and tongue.	Provide first aid to those affected by poisoning/irritation. If symptoms persist, take them to the nearest clinic or hospital.
E3	√				Strong acid catalysts (HCl and H ₂ SO ₄) are corrosive and can cause injury to practitioners.	Be careful when using strong acid catalysts and wear full PPE.	Provide first aid by running water over the wound. If bleeding continues, take the patient to the nearest clinic or hospital.

3. ANALYSIS / FINAL PRACTICAL							
E5. E6			√		When titrating free fatty acids on hydrolysis products with alcohol solvents, there is a	Ensure that alcohol does not come into direct contact with heat sources/flame sources. Be careful when using alcohol so that it does	Extinguish the fire with water or place a wet cloth over the source of the fire. If the fire is large

					risk of the alcohol catching fire and exploding.	not spill; you can use a funnel as a tool to help pour the alcohol.	enough, use a fire extinguisher to put it out.
C1 C3			√		When titrating free fatty acids on hydrolysis products, the solution is heated with an electric stove. Flaked wires can cause electric shock.	Check before use to ensure that the tool is in good condition.	Unplug the power source to stop the flow of electricity.

Process

Laboratory