Work Plan RE1 - Biodiesel Production in a Batch Reactor

Group B5
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1 Introduction

In this experiment, biodiesel will be produced in a batch reactor. Biodiesel is made from a transesterification reaction of soybean oil by methanol, where sodium hydroxide is used as a catalyst. The samples will be obtained in intervals, and separated to glycerine and biodiesel in a centrifuge. Further, the biodiesel will be analysed using gas chomatography to observe the progress of the transesterification reaction, where SBO goes to fatty methyl esters. The catalyst product selectivity and the reaction order will also be observed, and the conversion over time will be plotted.

2 Theory

2.1 Biodiesel

In recent years, biodiesel has gained attention as it can be used to reduce CO_2 emissions, because biodiesel does not add more to the carbon cyclus, while fossile fuel does that. Biodiesel can be produced from fat, from sources such as colza oil or soy beans. The boiling point and ignition temperature of fat are to high to be used as fuel without processing. One method for making biodiesel from fat is transesterification. Using methanol, the fat is transesterified into fatty methyl esters, which can be used as fuel. [1]

2.2 Reaction Mechanism

Figure 2.1 shows the reaction mechanism of the alkali-catalysed transesterification of vegetable oils. The figure is taken from the lab document. [2] The reaction consists of three reversible steps. First the catalytic base reacts with the alcohol, making an alkoxide and a hydrogenated base. In the next step a nucleophilic attack by the alkoxide at the triglyceride forms an intermediate, generating an alkyl ester and an anion of the diglyceride. In the final step the anion deprotonates the hydrogen base. The di- and monoglycerides are converted into a mixture of alkyl esters and glycerine using the same reaction mechanism. [2]

2.3 Kinetics in Batch Reactors

The mole balance for a batch reactor is:

$$F_{A0} - F_A + G_A = \frac{dN_A}{dt} \tag{2.1}$$

where F_{A0} is the initial flow of substance A in to the system, while F_A is the flow out of the system. G_A is the amount of substance A that has been generated in the reactor. $\frac{dN_A}{dt}$ is how much substance that has been accumulated in the system over a short period of time.

In a batch reactor, the flow into the system is the same as the flow out of the system, so F_{A0}=F_A. G_A can be written like this (where r_A is the reaction rate):

$$G_A = r_A * V = \frac{dN_A}{dt} \tag{2.2}$$

It is desirable to find the reaction rate, and since the volume is constant (V = V_0) the reaction rate is given by:

$$r_A = \frac{dN_A}{V_0 dt} = \frac{dC_A}{dt} \tag{2.3}$$

where C_A is the concentration of the substrate A.

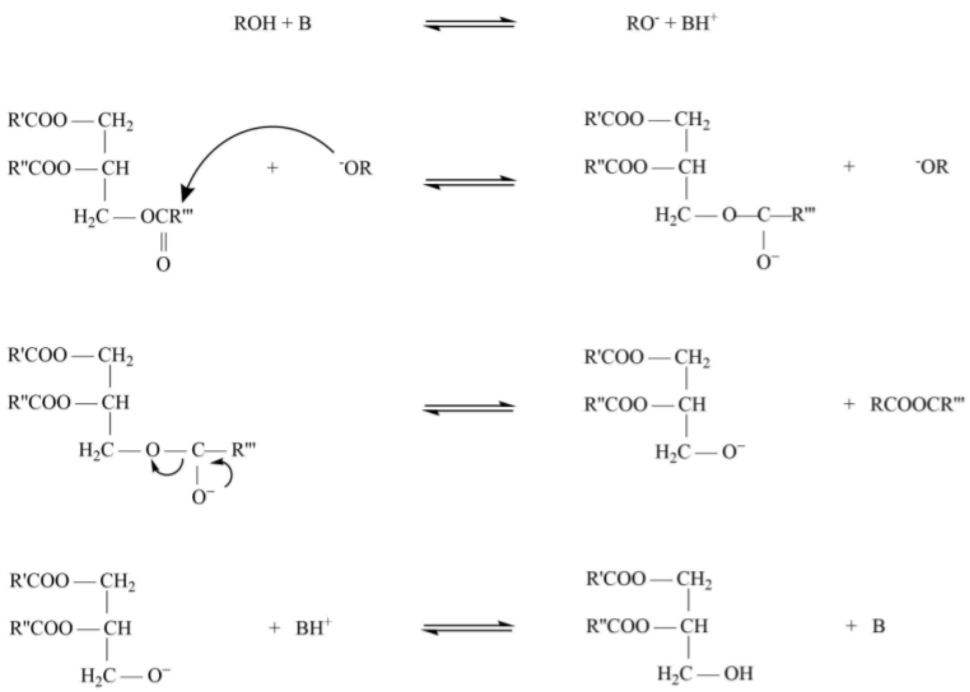

Figure 2.1: The reaction mechanism for the transesterification reaction [2]

3 Experimental Procedure

3.1 Apparatus

Figure 3.1 shows the setup of the reactor used during the experiment. The picture was collected from the lab document [2].

3.2 Execution

The experiment consists of two main parts: The preparation of biodiesel, and the analysis of the product.

3.2.1 Preparation of Biodiesel

- Measure out 279.3 mL of SBO, and pour it into the batch reactor. Add SBO first, and then the stirring rod, before fastening the lid of the reactor. Place the thermometer in one neck and close the last two necks that are not used with stoppers.
- Start stirring the reactor at 290 rpm. The water needs to be at the same level as the reactor solution before the heat of the water bath is turned on.
- In order to stop the reaction, the samples needs to be cooled, therefore: prepare an ice bath
- Measure out 70.7 mL MeOH in a beaker. Weight 1.65 g of NaOH and add the pellets to the beaker, and cover it with Parafilm immediately. The solution then needs to be stirred with a magnetic stirrer.
- When the temperature reaches 50 °C and the pellets are dissolved - the methoxide solution will be added using a funnel. When everything has been added, the start the stopwatch (this is the beginning of the reaction).

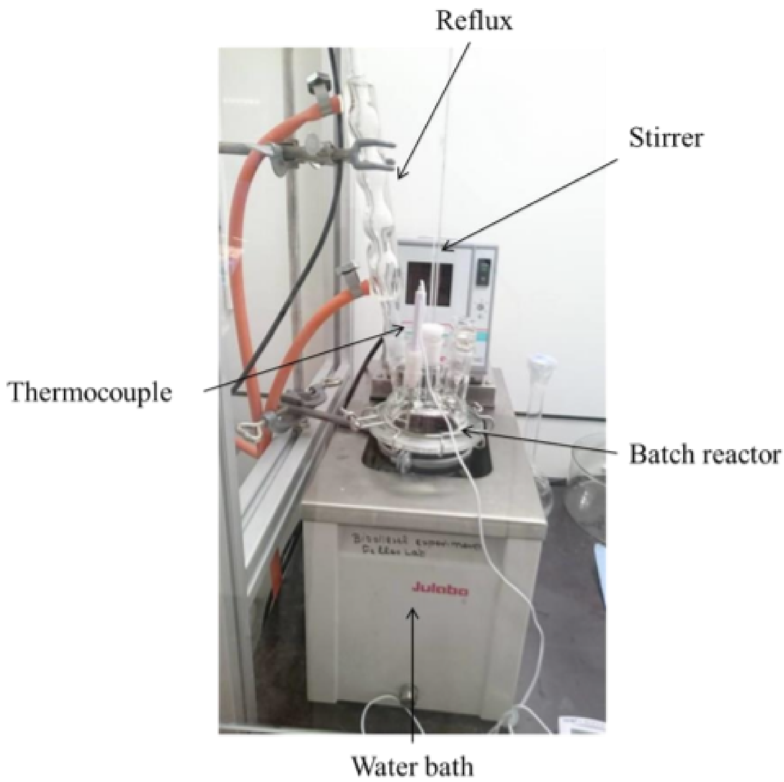

Figure 3.1: The set up for the experiment [2]

- After 3 minutes, the samples will be extracted (5mL). It is necessary to change the pipette tube for each sample. The time and temperature has to be monitored and recorded for each sample.
- Number centrifuge tubes from 1 to 12 and place the samples in them. Leave the tubes in the icebath. Shake slightly.
- Wipe off the centrifuge tubes when all the samples are collected, and place them in the centrifuge. Lastly, let the centrifuge run for 10 min at 4000 rpm.

3.2.2 Product Analysis

- By using a pipette: weigh approximately 250 mg of each samples in small glass vials. Only extract liquid from the upper layer.
- Add 5 mL IST to all of the samples.
- Transfer 1 mL of the sample solution to the GC vials. GC analysis will be performed by Kishore.

4 Health, Safety and Environment

Lab coat and glasses is always mandatory in the lab. There are some hazards associated with the chemicals used in this experiment, which makes some extra safety precautions necessary.

Methanol will be handled in this experiment. It can be toxic if it is swallowed, inhaled or in contact with the skin. Therefore it is important to wear protective gloves while handling methanol. It is also highly flammable, and causes damage to the organs, so it should be handled with care, and kept away from heat.

NaOH is corrosive on skin and eyes, so gloves are necessary.

The IST is dissolved in heptane. Heptane is flammable, can cause skin irritation and can be deadly if swallowed. It is also poisonous to aqueous environments. The heptane solution should be handled carefully. The solution will be kept under a fume hood, and handled with gloves.

The COVID-19 restrictions has been repealed, so it is not necessary to do much due to infection control. As a precaution, the lab space and equipment used during the experiment should be wiped before leaving the lab.

Trondheim, January 9, 2022

Karianne Høie, Erlend Sørlie

References

[1] Bjørn Pedersen. Biodiesel. https://snl.no/biodiesel, Last updated July 2021. Accessed: 2021-10-18.

[2] Kishore Rajedran. Felleslab-re1: Biodiesel production in a batch reactor. https://folk.ntnu.no/preisig/HAP_Specials/Felles_lab/Experiments/RE1_biodiesel.pdf, 2021.

A Data Tables

Table A.1 shows the assigned reaction conditions:

Table A.1: Reaction conditions

Reaction Condition	Value
Temperature [°C]	50
Stirring speed [rpm]	290
Mole fraction [MeOH/SBO]	6
C_{NaOH} [% w/w]	0.5
Total volume [mL]	350

Table A.2 shows relevant physical data for the compounds used or formed during the experiment [2].

Table A.2: Molar weight and density for the relevant compounds. [2]

Compound	Molar weight [g/mol]	Density [g/mL]
MeOH	32.04	0.792
NaOH	40.00	2.130
SBO	875.1	0.913
IST	270.5	0.853
BIOD	291.5	0.891
C16:0	270.5	-
C18:0	298.5	-
C18:1	296.5	-
C18:2	294.5	-
C18:3	292.5	-

B Calculations

B.1 Amount of Reactants and Catalyst

The molar fraction in Table A.1 gives,

$$\frac{n_{\text{MeOH}}}{n_{\text{SBO}}} = 6 \tag{B.1}$$

Where n_i is the number of moles of component i. Equation (B.1) can be rearranged into this expression:

$$n_{\text{MeOH}} = 6 \cdot n_{\text{SBO}} \tag{B.2}$$

The total volume is given in Table A.1, and can be expressed as,

$$V_{\text{tot}} = V_{\text{MeOH}} + V_{\text{SBO}} \tag{B.3}$$

Where V_{MeOH} and V_{SBO} are the added volumes of MeOH and SBO, respectively. The relationship between the volume and the number of moles of any compound, i, can be expressed using,

$$V_i = \frac{n_i \cdot M_i}{\rho_i} \tag{B.4}$$

Where M_i is the molar weight of the compound, and ρ_i is the density of the compound.

Inserting equations (B.2) and (B.4) into equation (B.3), inserting the values in Table A.2 and solving for n_{SBO}, gives:

$$n_{\text{SBO}} = \frac{V_{\text{tot}}}{6 \cdot \frac{M_{\text{MeOH}}}{\rho_{\text{MeOH}}} + \frac{M_{\text{SBO}}}{\rho_{\text{SBO}}}} = \frac{350}{6 \cdot \frac{32.04}{0.792} + \frac{875.1}{0.913}} \,\text{mol} = 0.2914\,\text{mol}$$

Rearranging equation (B.4), using n_{SBO} and the data from Table A.2, the necessary volume of SBO, $V_{\text{SBO}} = 279.3\,\text{mL}$.

Inserting V_{SBO} and V_{tot} into equation (B.3), the volume of MeOH is determined: $V_{\text{MeOH}} = 70.7\,\text{mL}$.

The amount of NaOH catalyst in the solution should be 0.5 %w/w, which gives the following relation:

$$m_{\text{NaOH}} = 0.005 \cdot m_{\text{tot}} \tag{B.5}$$

Where m_{NaOH} is the mass of NaOH and m_{tot} is the total mass of the solution.

The total mass is the sum of the mass of SBO, MeOH and NaOH. The mass of SBO and MeOH can be calculated using,

$$m_i = \rho_i \cdot V_i \tag{B.6}$$

The total volume of the solution can then be expressed as:

$$m_{\text{tot}} = m_{\text{SBO}} + m_{\text{MeOH}} + m_{\text{NaOH}} = \rho_{\text{SBO}} \cdot V_{\text{SBO}} + \rho_{\text{MeOH}} \cdot V_{MeOH} + m_{\text{NaOH}} \tag{B.7}$$

Inserting equation (B.5) into equation (B.7), the total mass of the solution becomes:

$$m_{\text{tot}} = \frac{0.913 \cdot 297.3 + 0.792 \cdot 70.7}{0.995}\,\text{g} = 329.1\,\text{g}$$

Inserting m_{tot} into equation (B.5), the required mass of NaOH is determined: $m_{\text{NaOH}} = 1.65\,\text{g}$. The calculated amounts of the reactants required in the experiment is shown in Table B.1.

Table B.1: The calculated amounts of the reactants required in the experiment.

Compound	Number of moles [mol]	Mass [g]	Volume [mL]
MeOH	1.7484	56.01	70.7
NaOH	-	1.65	-
SBO	0.2914	255.0	279.3

B.2 Weight Percentage of IST

The weight percentage of IST in the samples can be calculated using:

$$\text{wt\%}_{\text{IST}} = \frac{m_{\text{IST}}}{m_{\text{tot}}} = \frac{V_{\text{IST}} \cdot C_{\text{IST}}}{m_{\text{tot}}} = \frac{5\,\text{mL} \cdot 5\,\text{mg}\,\text{mL}^{-1}}{250\,\text{mg} + 5\,\text{mL} \cdot 5\,\text{mg}\,\text{mL}^{-1}} = 9.09\,\%$$