

## Production of fuel from rapeseed oil

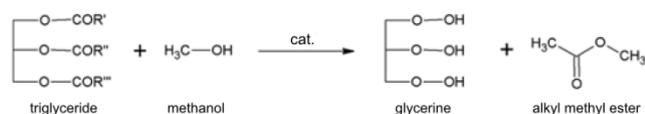
### Aims of the experiment

- To produce a fuel.
- To learn about regenerative energies and ecological balances.
- To understand transesterification as a type of reaction.

### Principles

Today, there is an ever-growing need for energy sources. However, as fossil sources of energy are limited and the increasing need results in a shortage, equivalent renewable energy sources are being searched for. The renewable energy sources also include environmentally friendly fuels with a CO<sub>2</sub> balance as neutral as possible.

Biodiesel is one of these renewable fuels and is produced from fats and oils through transesterification. Fats are triglycerides, which means that they are esters consisting of glycerine and three fatty acids. The triglycerides are converted to alkyl esters by catalysis with ethanol or methanol. Here, transesterification is most common with methanol.



This transesterification is necessary so that the biodiesel

possesses properties that are similar to those of diesel fuel. Rapeseed oil itself is not suitable as a fuel, as it is too viscous and therefore would not be optimally transported into the engine.

Mostly oil seeds or other oil-containing parts of plants are used as raw materials. Used this way, the fuel achieves a neutral CO<sub>2</sub> balance, as only as much CO<sub>2</sub> is released upon combustion as the plant has absorbed during photosynthesis. In Europe, biodiesel is produced mainly from rapeseed, which has an oil content of 40 - 45 %.

However, the positive environmental balance of biodiesel produced from rapeseed is questioned, as large areas of cultivable land are necessary and a large quantity of fertilisers must be used. Because of this, the environmental balance will ultimately be not much better than that of fossil fuels. Also, the total demand for fuel in Germany cannot be met with biodiesel, as the required area of arable land is not available for this.



Fig. 1: Set-up of the experiment for the production of fuel from rapeseed oil.

## Risk assessment

Sodium hydroxide is a corrosive substance. Wear protective clothing (lab coat, protective goggles, gloves).

Methanol is a poisonous substance. Wear protective clothing (lab coat, protective goggles, gloves) during use. On skin contact, it is imperative to wash with plenty of water and soap.

Sodium hydroxide pellets	
 <p><b>Signal word:</b> <b>Hazard</b></p>	<p><b>Hazard statements</b></p> <p>H314 Causes severe skin burns and eye damage.</p> <p>H290 May be corrosive to metals.</p> <p><b>Precautionary statements</b></p> <p>P280 Wear protective gloves/protective clothing/eye protection/face protection.</p> <p>P301+ P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.</p> <p>P309+P310 If on skin: Wash with soap and water.</p> <p>P305+P351+P338 IF IN EYES: Rinse continuously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.</p>
Methanol	
   <p><b>Signal word:</b> <b>Hazard</b></p>	<p><b>Hazard statements</b></p> <p>H225 Highly flammable liquid and vapour.</p> <p>H331 Toxic if inhaled.</p> <p>H311 Toxic in contact with skin.</p> <p>H301 Toxic if swallowed.</p> <p>H370 Causes damage to organs.</p> <p><b>Precautionary statements</b></p> <p>P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.</p> <p>P233 Keep container tightly closed.</p> <p>P280 Wear protective gloves/protective clothing/eye protection/face protection.</p> <p>P302+P352 If on skin: Wash with soap and water.</p>

## Equipment and chemicals

1	Magnetic stirrer with hotplate.....	66 8471
1	Stirring magnet rods 25 mm x 6 mm diam. ....	666 851
1	Stirring magnet rods 15 mm x 5 mm diam. ....	666 850
1	Test tube DURAN, 20 x 180 mm .....	602 004
1	Test tube holder, wooden, 22 mm diam. ....	667 053
1	Beaker, DURAN, 250 ml, squat.....	664 103
1	Beaker, Boro 3.3, 100 ml, squat .....	602 022
1	Stand base, V-shaped, small.....	300 02
1	Stand tube 300 mm x 10 mm diam.....	608 050
2	Bosshead S.....	301 09
2	Universal clamp 0...80 mm .....	666 555
1	Stirring thermometer, -30...+110 °C/1 K .....	382 21
1	Measuring cylinder 50 ml with plastic base ...	665 753
1	Dropping pipette, 150 x 7 mm, set of 10.....	665 953
1	Rubber bulbs .....	665 954
1	Graduated pipette 2 ml.....	665 995
1	Graduated pipette 5 ml.....	665 996
1	Pipetting ball (Peleus ball).....	666 003
1	Rubber stopper, solid, 19-24 mm diam.....	667 257
1	Rubber stopper, one 7 mm hole, 19-24 mm diam. ....	667 258
1	Glass tube 300 mm x 8 mm diam.....	665 204
1	Petri dish, 100 mm diam.....	664 183
1	Methanol, 250 ml .....	673 2700
1	Sodium hydroxide, 100 g.....	673 68001
Also required:		
Rapeseed oil		
Cardboard		

## Set-up and preparation of the experiment

### Set-up of the apparatus

1. The experiment is set up as shown in Fig. 1.
2. Fix the stand tube in the stand base.
3. Fix the thermometer and the test tube to the stand tube using a bosshead and universal clamp in each case.
4. Fill the 250 ml beaker with water, place it on the hotplate and under the test tube so that the test tube and thermometer are immersed.

### Preparation of the experiment

1. For the experiment, weigh 0.15 g of sodium hydroxide into a beaker.
2. Measure 50 ml of methanol with a measuring cylinder and add to the sodium hydroxide pellets. The sodium hydroxide must dissolve completely in the methanol.  
*Note: It is important that everything is completely dissolved, as the reaction cannot run optimally otherwise. Insufficient sodium methanolate would then be formed.*
3. Place 2 ml of rapeseed oil and a stirring magnet into a test tube.
4. During the weighing and dissolving process, the water bath can be heated to 60 - 65 °C. Check the temperature with the stirring thermometer.

## Performing the experiment

### Synthesis of biodiesel

1. When the temperature of the water bath is reached and the sodium hydroxide is completely dissolved, add 4 ml of the methanolic solution to the rapeseed oil.
2. Insert the glass tube into the stopper as a reflux cooler.
3. Close the test tube with the stopper and immerse it in the water bath.

4. During heating with constant stirring, a cloudy emulsion should form at first.

*Note: However, this cloudy emulsion dissipates again after a few minutes.*

#### **Purification of the experimental preparation**

1. When the solution is completely clear again, it can be added to another test tube filled three quarters full with water.
2. Close the test tube with a stopper and shake vigorously.
3. A recognisable phase separation should result within five minutes.
4. The upper phase can then be carefully removed using a Pasteur pipette and placed in a third test tube.
5. This phase consists of biodiesel.

#### **Comparison between rapeseed oil and biodiesel**

There are two possibilities available to check the identity of the product after the experiment. On the one hand, the flow speeds of the starting material and product can be compared with each other. On the other hand, a simplified comparison of the viscosity of both substances is possible.

A Petri dish is necessary for the comparison of the flow speeds. Add to this one drop each of rapeseed oil and biodiesel at the same level. Then hold the Petri dish at an angle and observe the flow speed of both drops.

For a quick comparison of the viscosity of rapeseed oil and biodiesel, a piece of cardboard is needed. Place one drop each of both substances onto this as simultaneously as possible. Then observe which of the two substances passes through the cardboard more quickly due to having a lower viscosity.

#### **Observation**

In the conversion of rapeseed oil with methanolic sodium hydroxide solution, a cloudy emulsion forms at first. After a few minutes, two phases form from the emulsion. The upper

phase contains the biodiesel, which has a lower density than water and therefore floats on top. The lower phase mainly contains glycerine.

The preparation from the experiment can be purified with water by shaking out, and the upper phase removed as the product.

#### **Result**

In this experiment, biodiesel has been produced from the renewable raw material rapeseed oil. Using sodium hydroxide as a catalyst, the fatty acid esters contained in the biodiesel have been converted with methanol to methyl esters.

Excess methanol and glycerine are removed from the solution using water. Contaminants such as soaps also dissolve in the water. Phases are formed due to the different densities. The upper phase contains the biodiesel, which has a lower density than water and therefore floats on top.

The resulting biodiesel can be compared in two brief experiments with the rapeseed oil as the starting material. In the one version, the flow speed is compared. One drop each of biodiesel and rapeseed oil are added to a Petri dish. If this is held at an angle, it can be observed that the biodiesel has a higher flow speed relative to the rapeseed oil.

The second possibility is a comparison of the viscosity. For this, one drop each of both substances is placed on a piece of cardboard as simultaneously as possible. Here, the biodiesel passes through the cardboard more quickly compared with the rapeseed oil due to its lower viscosity.

Further analyses of the biodiesel produced are carried out in the experiment C 5.4.1.2.

#### **Cleaning and disposal**

The biodiesel can be collected in an appropriately labelled container and stored for further experiments. The aqueous phase must be disposed of in the organic solvent waste, as it contains residues of methanol.