Activity 1D – Biodiesel production

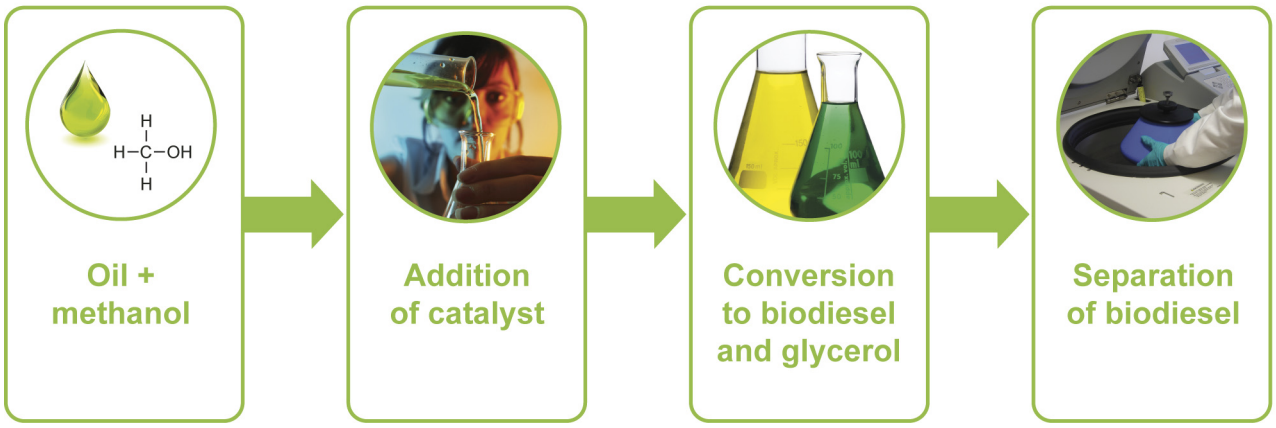
Learning outcomes: By the end of the session students should be able to:

- Describe the techniques used to produce biodiesel.
- Carry out the conversion of vegetable oil to biodiesel.
- Discuss the efficiency of biodiesel production and the uses of the by-products.

Keywords Bioenergy, biofuel, biodiesel, sustainable, renewable, biomass, yield, catalyst, methanol, glycerol, maize, oilseed rape, soya, potassium hydroxide, centrifuge, transesterification.

Background

In order to convert oil to biodiesel it is mixed with methanol, or occasionally ethanol, and a catalyst (potassium hydroxide) added to speed up the reaction. This is a transesterification reaction that produces biodiesel and glycerol. Biodiesel is less dense than glycerol and the two products are separated by gravity or using centrifuges. The biodiesel can be used to replace regular diesel or mixed with regular diesel in varying concentrations, while glycerol can be used in soap and cosmetics. The potassium hydroxide (KOH) catalyst increases the rate of reaction but does not increase the yield. Most of the KOH separates out into the lower glycerol layer and any remaining in the biodiesel is removed by washing with water.



This activity is based on the ones published by CLEAPSS®: Making Biodiesel. www.cleapss.org.uk and the Gatsby Science Enhancement Programme (SEP).

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Age Range: This activity is suitable for GCSE and A-level students.

Duration: 60 minutes.

Suggested prior knowledge: It is recommended that you elicit the existing student knowledge of fuels, properties of gases and liquids, catalysts and health and safety.

What you will need

- Test tubes and stoppers or 15 ml centrifuge tubes
- Test tube racks
- Centrifuge (a hand centrifuge can be used effectively)
- Cooking oil or oil extracted and filtered from an oil extraction (activity 1B)
- Methanol (VERY TOXIC and EXTREMELY FLAMMABLE)
- Potassium hydroxide (CORROSIVE)
- 5% w/w potassium hydroxide solution in methanol (5 g potassium hydroxide per 50 ml of methanol)
- Access to a fume cupboard
- Magnetic stirrer and stirring fleas
- Conical flask, 250 ml
- Conical flask, 100 ml
- Measuring cylinders 100 ml
- Disposable nitrile gloves
- Safety goggles

Optional

- Balance
- Separating funnel
- Dropping pipettes
- Distilled water

Health and Safety

It is essential that eye protection is worn, preferably goggles rather than safety spectacles. Nitrile gloves should be worn.

NOTE: when preparing the catalyst a fume cupboard is required in addition to goggles.

This experiment involves Methanol (VERY TOXIC and EXTREMELY FLAMMABLE) and potassium hydroxide (CORROSIVE). Methanol is the most effective solvent for use in this experiment but extra care needs to be taken to ensure that students wear Personal Protection Equipment (PPE) and act sensibly during this procedure. CLEAPSS® do not recommend using sodium hydroxide instead of potassium hydroxide because it has a lower solubility in ethanol. Consult technicians at a school prior to carrying out this activity as it is far safer to have the KOH methanol mixture made up in school in small aliquots already prepared in stoppered test tubes for students. If the reagents are taken into school or used at an event ensure that the correct risk assessments have been completed, the hosts have been informed of the planned activity and that the correct regulations concerning transport of chemicals are adhered to. The guide - Transporting chemicals for lecture demonstrations & similar purposes, Royal Society of Chemistry (RSC), January 2008-suggests that small volumes can be transported safely if clearly labelled with LQ (limited quantity) notices.

NOTE: Provide clear instructions to the students NOT to shake the test tubes as the methanol may squirt out.

Any contact of the methanol solution or biodiesel mixtures with the skin should be washed off under a tap straight away.

The volume of methanol used in the preparation of the catalyst and the activity pose a low risk of exposure by vaporisation. Methanol and biodiesel mixtures are highly flammable and should be kept away from naked flames

For further information read the CLEAPSS® Guidance PS 67-10 (Making bio-diesel), Recipe book RB71 (Potassium hydroxide), RB102 (Testing for organic functional groups) and Hazcards 40B (Methanol), 40C (Carbohydrates), 45 and 46 (Hydrocarbons).

If using a centrifuge ensure that the centrifuge tubes are balanced and that the tubes used for centrifugation are sealed.

Method

NOTE: Steps 1 and 2 should be carried out by a properly qualified adult in a fume cupboard. See the Health and Safety instructions for further information.

1. Prepare the catalyst. A 5% w/v potassium hydroxide (KOH) solution should be prepared with methanol by adding 5 g of KOH to 100 ml of methanol in a conical flask or beaker on a stirrer. Wait until all the potassium hydroxide has dissolved.
2. 1 ml or 1.5 g of the methanol KOH solution should be aliquoted into test tubes or centrifuge tubes and sealed prior to the activity. If any of the solution remains it should be stored in an airtight borosilicate glass bottle and clearly labelled, adding the hazard symbols CORROSIVE, TOXIC and EXTREMELY FLAMMABLE.
3. Students can add 10 ml or 10 g of vegetable oil to a test tube or 15 ml centrifuge tube.
4. Add the contents of the tube containing the methanol / potassium hydroxide catalyst to the tube containing the vegetable oil and ensure the tube is properly sealed.
5. In order to carry out the conversion reaction the tubes should be carefully and slowly inverted over 30 times to ensure adequate mixing.
6. Students should carefully observe the contents of the test tube. The biodiesel separates out in the top layer while a lower layer of glycerol gradually forms.
7. Students should label the tubes with the contents and their names.

Students will be able to observe a reaction almost immediately on mixing the oil and catalyst. The oil will quickly become less viscous. However, it is better to prepare biodiesel in one lesson, store it in labelled test tubes to allow the layers to separate fully, and perform any follow-up tests in the next lesson. If the products of the reaction are to be tested it is best to wait at least 24 hours for full separation or centrifuge the solutions.

Disposable pipettes can be used to separate the two products of the reaction by siphoning off the top biodiesel layer and transferring it to another test tube or centrifuge tube. Alternatively a separating funnel can be used to run out the lower layer of glycerol, leaving the layer of biodiesel behind. If a centrifuge is used to separate the products it is recommended that the mixture is transferred to microcentrifuge tubes using disposable pipettes and a small bench top centrifuge is used. Ensure that the centrifuge is balanced. Care is then needed to separate the two layers and a fine-tipped disposable pipette is recommended.

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Extension activities

Improve the quality of the biodiesel by carrying out a further washing step to meet the standards required for use in vehicle engines. Impurities in biodiesel cause problems in modern and high-end car engines and it is important to 'wash' the biodiesel prior to use. This can be carried out by the students by adding an equal volume of distilled water to the biodiesel, mixing the solution and repeating the separation procedure.

Calculate the yield of biodiesel. Have students weigh the oil, methanol and resultant biodiesel. Weigh the test or centrifuge tube prior to adding the oil, after adding the oil and after adding the methanol. Once the biodiesel and glycerol layers are completely separated the upper biodiesel layer can be carefully removed with a disposable pipette and added to a weighed container. Calculate the yield: (weight of biodiesel ÷ weight of oil and methanol) x 100 = % yield.

Compare the burning qualities of the biodiesel and vegetable oil. The biodiesel, oil from activity 1B, sugar from activity 1E and ethanol from activity 1G can be collected and tested for their combustion energy – see Gatsby SEP: Biofuels activity A7 'How much energy is released when a fuel burns?' or 'Energy values of food' from Practical Chemistry www.practicalchemistry.org/experiments/energy-values-of-food,225,EX.html. These activities should be carried out in a fume cupboard.

Compare the viscosity of the biodiesel and vegetable oil. See activity 1C Oil viscosity.

Test the biodiesel for saturation. For further details see CLEAPSS® Guidance PS 67-01 (Testing for unsaturation), 'Unsaturation in fats and oils' from Practical Chemistry www.practicalchemistry.org/experiments/unsaturation-in-fats-and-oils,227,EX.html or SEP Biofuels activity A5: Saturation of fuels.

Discussion activities about the practicalities of the technique used on an industrial scale, the sources of feedstocks and economic viability of producing biodiesel to replace fossil fuels.

Biodiesel UpD8 activity www.upd8.org.uk/activity/256/Biodiesel.html

Suppliers

Vegetable cooking oils can be obtained from supermarkets or local shops.

The laboratory equipment should be available in most secondary schools science departments. A small benchtop centrifuge can be used with smaller volumes.

A microcentrifuge suitable for school use can be obtained from National Centre for Biotechnology Education (NCBE) www.ncbe.reading.ac.uk/menu.html University of Reading, 2 Earley Gate, Whiteknights Road, Reading, RG6 6AU tel: 0118 9873743 fax: 01189 750140

Hand centrifuges can be obtained from Rapid www.rapidonline.com Severalls Lane, Colchester, Essex, C04 5JS tel: 01206 751166 fax: 01206 751188

A 'Green chemistry: production of biodiesel' kit containing the oil, alcohol and catalyst for this activity is available from Rapid (see above).

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Further reading and links

CLEAPSS® Guidance PS 67-10 (Making bio-diesel).

Gatsby Science Enhancement Programme (SEP): Biofuels. 2009. www.sep.org.uk

Making Biodiesel pre-16, Royal Society of Chemistry http://media.rsc.org/Learning%20about%20materials/Materials%20Biodiesel%20Part%201.pdf

Making Biodiesel post-16, Royal Society of Chemistry http://media.rsc.org/Learning%20about%20materials/Materials%20Biodiesel%20Part%202.pdf

The Royal Society, January 2008. *Sustainable biofuels: prospects and challenges*, ISBN 978 0 85403 662 2. http://royalsociety.org/Sustainable-biofuels-prospects-and-challenges/

Nuffield Council on Bioethics, April 2011, *Biofuels: ethical issues* www.nuffieldbioethics.org/biofuels-0

Research groups

Professor Gillian Stephens, University of Nottingham, Process Intensification for Acceleration of Bio & Chemo Catalysis in Biorefining www.nottingham.ac.uk/engineering/departments/chemenv/people/gill.stephens

Dr Sohail Ali, Plymouth Marine Laboratory, Integrated approach to cost effective production of biodiesel from photosynthetic microbes www.pml.ac.uk/about_us/pml_people/sohail_ali.aspx

Professor Antoni Slabas, Durham University, An integrated approach to the cost effective production of biodiesel from photosynthetic microbes www.dur.ac.uk/biosciences/about/schoolstaff/academicstaff/?id=40

Dr Sean Murphy, CAB International, Impacts of tropical land use conversion to jatropha and oil palm on rural livelihoods and ecosystem services in India and Mexico www.cabi.org/default.aspx?site=170&page=1019&sid=1426

Professor Johnathan Napier, Rothamsted Research, Rational metabolic engineering of oilseed fatty acid composition www.rothamsted.ac.uk/Research/Centres/PersonDetails.php?Centre=CGI&PIID=137037

Dr Kerrie Farrar, Aberystwyth University, Understanding and exploiting the diversity of form in Miscanthus

Dr James Murray, Imperial College London, Investigation of Water Oxidizing Catalysis for Renewable Energy