**Worksheet**

**Option 1: The reflux reaction of vegetable oil by transesterification to prepare a sample of biodiesel**

This reaction looks at the process used to create biodiesel, a ‘greener alternative’ fuel to diesel. The process involves reacting a vegetable oil, whose main constituent is a triglyceride, with methanol (or ethanol) and a catalyst under reflux, to form long chain methyl esters – biodiesel.

**Safety:**

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| --- | --- | --- |
| Chemical name | Rough quantity per student | Hazards |
| Vegetable oil | 12 g | Not classified as hazardous |
| Sodium hydroxide (4 mol dm-3) | 1 cm3 | Corrosive (H290, H214, H318) |
| Methanol | 10 cm3 | Flammable (H225), Toxic (H301, H311, H331) Health Hazard (H370) |
| Biodiesel | 10 g | Flammable, treat as corrosive and irritant |

**Equipment per group/student:**

* 50 cm3 round-bottom flask (RBF)
* Anti-bumping granules
* Vegetable oil
* Heating mantle/hotplate stirrer
* Lab jack
* (Liebig) condenser and connecting tubes
* 100–1000 µL autopipette (or 10 cm3 syringe if autopipette unavailable)
* Methanol
* 4 mol dm-3 sodium hydroxide solution
* Boiling tube
* Clamp, stand and bosses
* Access to a 2 d.p. balance and cork ring
* Sample vial
* Optional: glass wool, plastic pipette, heat proof mat, matches

**Method:**

1. Place a cork ring onto a 2 d.p. balance. Stand a 50 cm3 round-bottom flask (RBF) in the cork ring and zero the balance.
2. Transfer into the RBF roughly 12 g of your chosen oil. Record the actual mass of oil used (to 2 d.p.).
3. Firmly clamp the RBF above a heating mantle, on a lab jack. Add a small number of anti-bumping granules to the RBF if you are using a non-stirring mantle. If you are using a hotplate stirrer, add an oval stirrer bar to the flask.
4. Use the lab jack to raise the heating source mantle up to the flask.
5. Connect the condenser tubes up and turn on the water slowly. It should be a slow trickle at most.
6. Slot the condenser into the flask. Get a teacher to check your equipment before proceeding to the next step.
7. Down the top of the condenser (using an autopipette or syringe) add 3.0 cm3 of methanol.
8. Turn on the stirring to around 450 rpm (if applicable) and turn on the heating to about 120°C.
9. Allow the reaction to start gently refluxing. Once this has occurred, use an autopipette to add 750 µl (0.75 cm3) of the 4 mol dm-3 NaOH solution down the top of the condenser.
10. Leave the reaction to reflux (with stirring or your anti-bumping granules) for ten minutes. The reaction is ‘refluxing’ when the solution is boiling, and condensed vapour is steadily dripping off the condenser back into the flask.
11. After ten minutes, turn off the heat (but leave stirring) and lower the heating mantle away from the flask using the lab jack screw.
12. Once cool enough to handle, pour the contents of the flask into a gently clamped boiling tube (using a clamp rather than a test tube rack makes the next step easier).
13. Weigh an empty clean sample vial. Carefully using a plastic pipette, transfer the top layer (biodiesel) to the vial and then reweigh the vial. Determine and record your mass yield of biodiesel.
14. If your teacher allows you can compare the flammability of the biodiesel to the original oil, by using a plastic pipette to drop some (~0.5 cm3) onto some glass wool on a heatproof mat and setting it alight. You should see how much more flammable (easy to ignite, cleaner burning) the biodiesel is compared to the original oil.

**Option 2: The base hydrolysis of an ester, using reflux**

This reaction uses reflux to hydrolyse (add water across) an ester bond, breaking the ester bond and creating a salt and ethanol. Optional further practical steps involve acidifying the salt to precipitate out the carboxylic acid product and collecting the impure product under vacuum filtration.

**Safety:**

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| --- | --- | --- |
| Chemical name | Rough max quantity | Hazards |
| Sodium hydroxide (2 M) | 50 cm3 | Corrosive (H290, H214, H318) |
| Hydrochloric acid (2 M) | 50 cm3 | Corrosive (H290, H315, H319), Irritant (H335) |
| Ethyl benzoate | 2 cm3 | None listed |
| Benzoic acid | 2 g | Organ Toxicity (lungs, H372), Corrosive (H315, H318) |
| Ethanol | 2 cm3 | Flammable (H225), Irritant (H319), Toxic (H302, H371) |

**Equipment per group/student:**

* 100 cm3 round-bottom flask (RBF)
* Anti-bumping granules
* Heating mantle/hotplate stirrer
* 5 cm3 graduated cylinder
* Ethyl benzoate
* Lab jack
* (Liebig) condenser
* 50 cm3 graduated cylinder
* 2 mol dm-3 sodium hydroxide

**Additional equipment for isolation of solid (impure) product:**

* 250 cm3 conical flask
* 50 cm3 graduated cylinder
* 2 mol dm-3 hydrochloric acid
* Access to an ice bath (and clamps)
* Vacuum filter (Buchner flask and filter)
* Filter paper

**Method – refluxing:**

1. Clamp a 100 cm3 round-bottom flash (RBF) around halfway up your clamp stand. Add anti-bumping granules (if using a non-stirring mantle), or a stirrer bar (if using a hotplate stirrer) to the flask. Then, using a graduated cylinder, add 2 cm3 of ethyl benzoate to the flask.
2. Raise the level of your heating mantle using a lab jack, so it sits comfortably below the flask.
3. Connect the condenser tubes up and turn on the water slowly. It should be a slow trickle at most.
4. Using a graduated cylinder add 30 cm3 of 2 mol dm-3 sodium hydroxide to the flask.
5. Slot the condenser into the top of the flask. Get a teacher to check your equipment before proceeding to the next step.
6. Turn on the stirring (if applicable) and set the temperature to 160°C.
7. Allow the reaction to start refluxing gently boiling. The reaction is ‘refluxing’ when the solution is boiling, and condensed vapour is steadily dripping off the condenser back into the flask.
8. Leave to reflux for 30 minutes.
9. After 30 minutes, turn off the heating (but leave stirring) and lower the heating mantle away from the flask using the lab jack screw.

Only move on to the next steps if instructed to do so by your teacher.

**Method – isolation of solid (impure) product:**

1. Clamp a 250 cm3 conical flask containing 50 cm3 of 2 mol dm-3 HCl in an ice bath.
2. Once the glassware is cool enough to handle, remove the condenser from the top of the flask and turn off the water.
3. Remove the RBF from the stand, using the clamp as a handle (unscrew the clamp from the stand).
4. Pour the hot contents of the RBF carefully into the clamped 250 cm3 conical flask containing the acid to reacidify the mixture.
5. Allow to cool to ice cold for about five minutes. Occasionally swirl.
6. Filter and roughly dry the precipitate from the liquid using vacuum filtration. You may rinse the conical flask with a small amount (<5 cm3) of cold water if needed.