PREPARATION OF BIODIESEL

TECHNIQUES REQUIRED : Reflux apparatus, extraction, drying agents, yield calculation

EXPERIMENTAL PROCEDURE Work in pairs – **hand in individual reports***

Before the transesterification, the crude starting material (i.e. waste vegetable oil) must be filtered to remove any solid matter (food debris) and dried to remove residual water. This is done by adding anhydrous magnesium sulfate (as a drying agent) to the oil, then stirring the mixture and vacuum filtering the oil (this part of the experiment has been done on a large scale by the Chem 353 laboratory technician prior to the start of the laboratory period).

- **Methanol is flammable and toxic.**
- **Potassium hydroxide can cause skin burns. Avoid skin contact.**
- **Ethanoic acid is corrosive and a lachrymator.**
- **Work in the fumehood.**

The glassware components of the reflux apparatus and the Erlenmeyer flask need to be clean and dry. If they have recently been washed with water and are still wet, then rinse all the glassware with a little methanol before use.

In the fumehood, set up a hot plate stirrer with a metal heating block (check the fit size for your 100 mL round bottomed flask). Make sure the power cords are well away from the heated surface. Don't turn the power on yet.

Weigh out approximately 50 g of waste vegetable oil in a graduated cylinder. Record the volume of oil, since this is what will be measured for the product volume yield. Pour the oil into a 100 mL round-bottom flask and add a stirrer bar. Secure the round bottom flask in the metal heating block using a clamp to the fumehood racking (don't over tighten the clamp on the glassneck of the flask, but make sure it secures the flask in an upright position).

Calculate the amount of methanol needed in the experiment so that there is approximately a 10:1 molar ratio of methanol : oil (assume that the molar mass of the oil is 1000 g/mol and given the density of methanol is 0.792 g/mL: the amount of methanol should be about 20 mL), check your calculation with your TA before proceeding. Add the methanol to a 50 mL Erlenmeyer flask.

The amount of base catalyst (KOH) needed is 1% by weight of the oil used. Calculate the amount of KOH required (check with your TA) and then measure this amount and quickly transfer this into the methanol in the Erlenmeyer flask. Cover the solution with a watch glass. Swirl the solution until all of the KOH is dissolved. If necessary, use heat to help the dissolution.

Once all of the KOH is dissolved, add the methanolic KOH solution to the oil in the round bottomed flask. Complete the reflux apparatus set up by connecting water hoses to the condenser and inserting the condenser into the round bottom flask joint. Get your TA to check the set up. Turn on the stirring (enough to get the bar spinning consistently) and the heat to about 150. Bring the oil mixture to a boil (monitor and adjust the heat control as necessary) and then gently heat (*i.e.*enougb to maintain a steady reflux) for an hour.

After the reflux period, remove the reflux apparatus from the heat source and let it cool to a manageable temperature, use an ice bath to help cool the flask if needed. Once cool, pour the reaction mixture into a separatory funnel. Two layers should appear in the separatory funnel: a lighter layer (top), which contains the product biodiesel methyl esters, and a darker layer (bottom), which contains glycerol and unreacted oil. Carefully drain the bottom layer from the separatory funnel.

To purify the product biodiesel methyl esters present in the top layer, first add 15 drops of conc. ethanoic acid (also known as glacial acetic acid) then swirl the mixture. Now wash the methyl esters by adding 20 mL of hot water and swirling. After letting this separate into two layers, remove the aqueous layer. Repeat the wash process twice more. Remove the biodiesel methyl esters from the separatory funnel and measure the final volume and weight of the product.

CLEAN UP

- Aqueous waste should be poured into the aqueous waste container.
- Biodiesel should be poured into the container specifically labelled for this.
- All other residues should be placed in the organic waste drum in the fumehood.

REFERENCES

1 "Organic Chemistry On-Line Learning Center", Ch 26, https://www.chem.ucalgary.ca/courses/351/Carey5th/Ch26/ch26-0.html.
M. Jones and S.A. Fleming, in "Organic Chemistry", Norton, 4th ed., 2010, Chapter 18, p. 895-7; 5th ed. 2014, Chapter 18, p. 896-9

2. Bucholtz, E.C., Biodiesel Synthesis and Evaluation , Journal of Chemical Education, **2007**, *84*, 296