Methods of pollution control and waste management

Experiment 24

Chemical recycling of poly(ethylene) terephthalate (PET)

Manual

Department of Chemical Technology

The aim of this experiment is to gain knowledge of plastic recycling methods and to develop skills in performing chemical recycling of waste polyethylene terephthalate, the comparison of glycolysis and hydrolysis reactions with respect to yield and environmental impact.

Equipment/Materials: two-necked round flask, reflux condensers, round bottom flask, Petri dishes, heating mantle, filtering flask with Büchner funnel, beakers, magnetic stirrer, melting point apparatus (Figure 1), FT-IR spectrophotometer (Figure 2), PET bottle waste, ethylene glycol, zinc acetate, sodium hydroxide, hydrochloric acid (37%), ethanol (96%), calcium acetate.

Figure 1. Melting point apparatus.

Figure 2. Shimadzu FT-IR model 8400S spectrophotometer.

Experimental schedule

1. Glycolysis of PET

2. Hydrolysis of PET in a basic environment

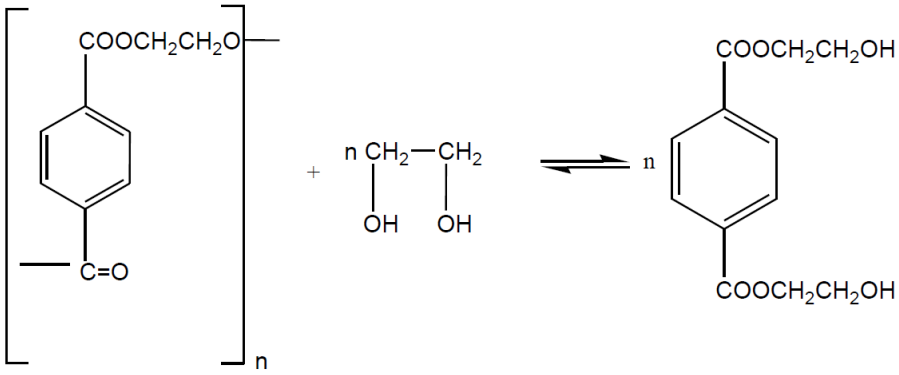
3. Products separation

4. Purification of the products

5. Products analysis (FTIR spectrum of terephthalic acid and melting point of BHET measurements).

1. **Glycolysis.**

Glycolysis of PET can be illustrated by equation (1)



(1)

Procedure:

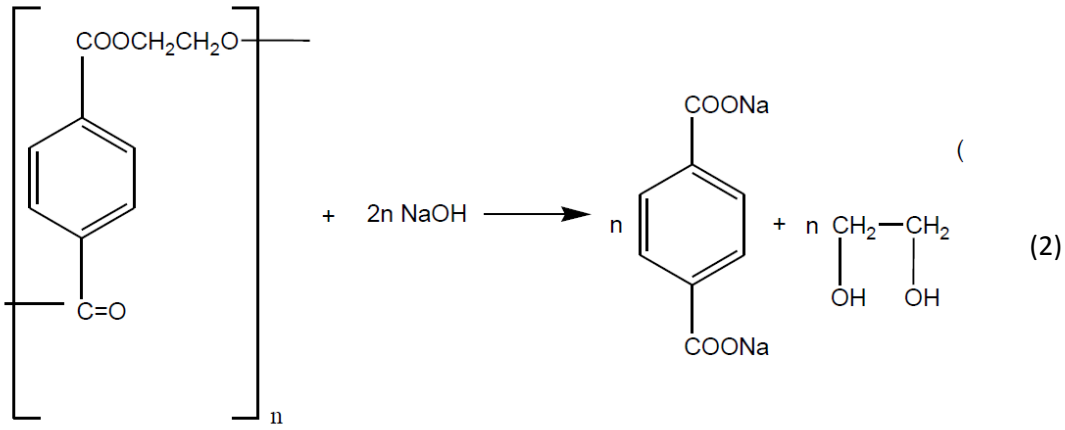
1. Cut up the plastic bottle made of PET into small pieces with scissors and then make a weighed portion of this material (about 10 g with accuracy 10 mg) and place in a two-necked flask, add 50 ml of ethylene glycol 0.5 g zinc acetate and several molecular sieves, place a thermometer (with a range 0-250^{0}C) in the side neck of the flask
2. Adjust the reflux condenser, open the flow of cooling water in the condenser, turn on the heating mantle and heat the reaction mixture under reflux for 1 hour
3. Observe the variations in temperature during the reaction, note the range of temperature changes in the results sheet
4. After 1 h remove the heating mantle and leave the laboratory set to cool down
5. Transfer the reaction mixture into the beaker with crushed ice (ca. 100 g), stir the mixture with a glass rod. After a few minutes a white precipitate should appear.

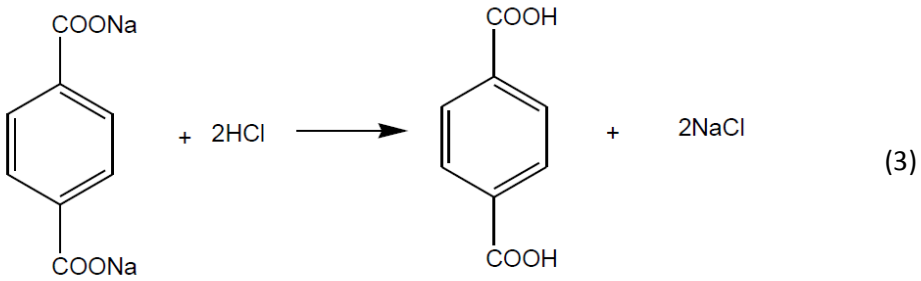
6. Assemble a vacuum filtration kit and filter the solid on the Büchner funnel under reduced pressure and wash it with 40 ml of water with ice
7. Remove the filtrate, clean the flask and adjust the Büchner funnel with residue, wash the precipitate with 150 ml of hot water, stir carefully with a glass rod and filter under reduced pressure
8. Transfer the filtrate (from point 7) into a beaker with 100g of crushed ice, after a few minutes white crystals (the main product) should appear
9. Filter the crystals on a Büchner funnel, under reduced pressure and transfer the obtained residue to a weighed Petri dish and dry in a laboratory dryer for 2 hours
10. Weigh the product, calculate the percentage yield of the reaction

During the reaction course measure the melting point of the product of glycolysis obtained by previous groups of students.

2. Hydrolysis of waste PET in a basic environment

Hydrolysis of PET in a basic environment can be illustrated by equations (2) and further reaction to terephtallic acid by equation (3):





Procedure:

1. Pour 90 ml of distilled water into a one-necked round-bottom flask, add 18g NaOH and stir with a magnetic stirrer to dissolve then add 45 ml of ethanol
2. Cut up the plastic bottle made of PET into small pieces with scissors and then make a weighed portion of this material (about 15 g with accuracy 10 mg) and place the prepared material in the flask
3. Adjust the reflux condenser, open the flow of cooling water in the condenser, turn on the heating mantle and heat the reaction mixture under reflux for 1 hour, Note: the reaction mixture should be stirring during the whole time of the reaction
4. After 1 h remove the heating mantle and leave the laboratory set to cool down, a white precipitate should appear during the reaction course
5. Filter the post-reaction mixture on the Büchner funnel under reduced pressure, discard the filtrate and wash the residue with 200 ml of distilled water, stir the mixture on the funnel with a glass rod
6. Transfer the obtained filtrate to the beaker
7. Add (carefully!) dropwise a concentrated hydrochloric acid to pH=6 using a glass pipette, the mixture should be stirred with a glass rod
8. Filter the precipitated white solid on a Büchner funnel under reduced pressure
9. Transfer the obtained residue to weighed Petri dish and dry it in laboratory dryer for 2 hours
10. Weigh the product and calculate the percentage yield of reaction

During the reaction course prepare a pellet from the mixture of the product of hydrolysis and KBr (3 mg :300 mg) by the means of the laboratory press (Figure 3). Record the FTIR spectrum of the product using a spectrophotometer (Figure 2).

Prepare, as described above a pellet from the mixture of commercially available terephtallic acid and KBr and record the FTIR spectrum of this sample. Compare the recorded spectra.

Figure 3. Manual hydraulic laboratory pellet press.