Chem 224 Exp. # 8: Polymer Preparation and Analysis FS2017

Experimental Procedure

Special Materials Needed:

3- six inch disposable test tubes
6- wooden applicator sticks (Form 3 stirring rods by taping pairs endwise to extend length)

Expt. 1 Condensation Polymers

In this experiment you will prepare both a linear polyester and a crosslinked polyester. Place 1 g of phthalic anhydride and 0.05 g of sodium acetate in each of two 6 inch disposable test tubes. To one tube add 0.4 mL of ethylene glycol and to the other add 0.4 ml of glycerol. Stir to dissolve. Clamp both tubes so that they can be heated simultaneously on a hot plate using the aluminum heat exchange block. Heat the tubes gently until the solutions appear to boil (water is eliminated during the esterification), then continue heating for 5 minutes.

Allow the two test tubes to cool and compare the viscosity and brittleness of the two polymers via the wooden applicator sticks. Record your observations. Note the order of polymer viscosity and brittleness. The test tubes cannot be easily cleaned and should be placed into the glass waste container in the waste hood.

Expt. 2 Addition Polymer

This preparation of polystyrene is mechanistically quite different from the preceding condensation experiment. Place about 4 mL of styrene monomer in a six inch disposable test tube and add 0.15 g of benzoyl peroxide.

CAUTION: Do NOT allow the peroxide to contact any paper during weighing as it is a strong oxidant. Also, keep the styrene under a hood as much as possible, as it is rather smelly.

Stir to dissolve. Clamp and heat the test tube in the aluminum block. Heat until the mixture turns yellow. When the color disappears and bubbles begin to appear, immediately raise the tube of styrene off of the hot plate, because the reaction is exothermic. After the reaction subsides, put the test tube of styrene back on the hot plate and continue heating it until the liquid becomes very syrupy. With a wooden applicator stick, draw out a long filament of material from the beaker. If this filament can be cleanly snapped after a few seconds of cooling, the polystyrene is adequately polymerized. If the filament does not break, continue heating the mixture and repeat this process until the filament breaks easily. Allow to cool and check the viscosity and brittleness with the wooden applicator stick. Record your observations. Comment on outcome: Compare & contrast the two procedures.

Prelab Questions:

1. Write the general equations for the structure of the linear and crosslinked polyesters formed in expt. #1.

2. Write the general equations and structures to form polystyrene from styrene and a peroxide.

3. What differences in functional group absorptions would be expected in the IR of the polyesters compared to the IR of the starting materials?