Exp. No. (2)

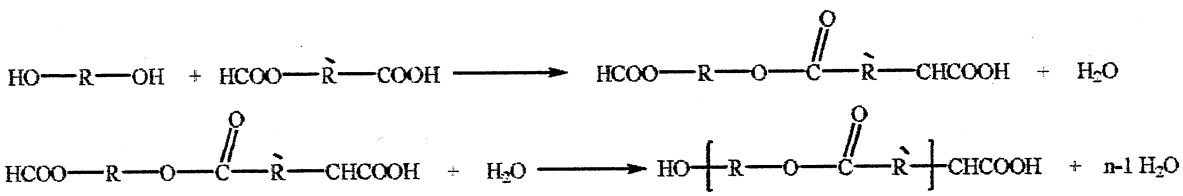
Preparation of Polyester by Condensation Polymerization

Introduction

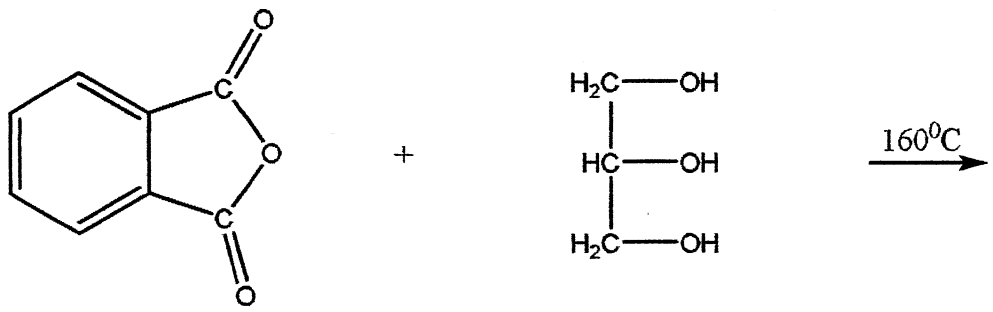
Step-growth polymerization is a random reaction of two molecules that may be any combination of a monomer, oligomer, or a longer-chain molecule, and usually small molecules like H_2O, CH_3OH, HCl, etc. are eliminated. High-molecular-weight polymer is formed in the early stages

An example of this type of polymerization is the formation of laminating resin polyester (Glyptal) from the reaction of glycerol and phthalic acid.

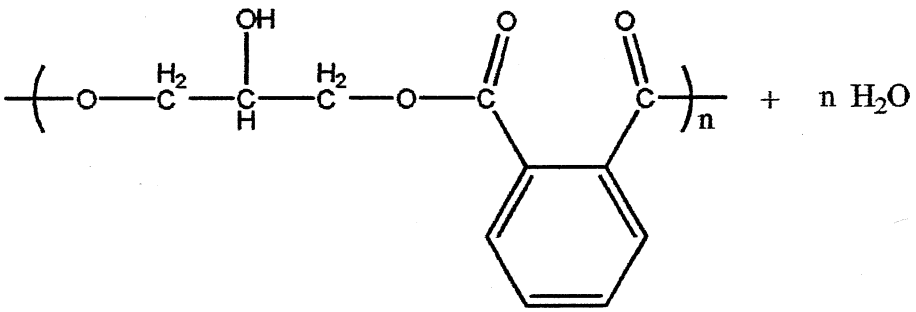
Polyesters are being commercially produced from the combination of two different monomers, one have two (or more) hydroxyl groups and the other have two (or more) carboxyl groups:



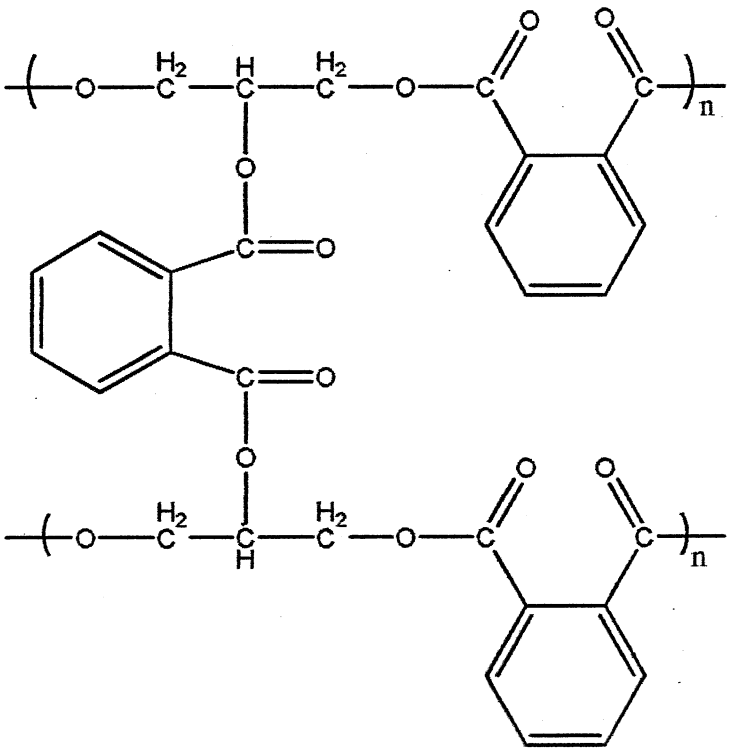
The reaction between glycerol and phthalic anhydride is basically esterification, and H_2O molecule is eliminated.



Phthalic anhydride Glycerol



Linear poly ester



Cross linked poly ester

Apparatus

1. Pyrex test tube
2. Thermometer
3. Crucibles

Reagent and materials

1. Phthalic anhydride
2. Glycerol
3. Conc. Sodium hydroxide solution
4. Conc. Sulfuric acid
5. Solvent (chloroform, acetone and ethanol)

Procedure

1. 2.5g of phthalic anhydride and 1g of glycerol are mixed in dry Pyrex tube with stopper and thermometer.
2. Heat slowly with careful stirring mixture inside the hood.
3. Rise the temperature to about 160°C and keep the reaction for 15 min at this temperature until all the solid is dissolved.
4. Some drops of produced polymer is pull in two crucibles for analysis.
5. The temperature of polymerization reaction is then rised slowly until it become 200-240°C where cross-linked polymer is formed.
6. Another few drops of product cross-linked polymer is pull in another two crucibles for analysis.

7. The collected drops of polymer samples, two crucibles contain linear and two crucibles contain cross-linked polyester are tested as following:

   a) *Conc. Sodium hydroxide test:*

      3ml of conc. Sodium hydroxide is added to one linear polyester crucible and another 3ml for one cross-linked polyester crucible.

   b) *Acid test:*

      3ml of conc. H_2SO_4 is added to one linear polyester crucible and another 3ml for one cross-linked polyester crucibl.

   c) *Melt test:*

      Heating the prepared polymer samples (linear and cross-linked polyester) on a heater and recorder your notes.

   d) *Solubility test:*

      3ml of one of the following organic solvents as, chloroform, acetone, CCl_4, ethanol, or any available organic solvent in the lab.

8. Record your notes and obtained results.