

ACTIVATION ENERGY DETERMINATION OF A NOVEL LIQUID CRYSTALLINE COPOLYESTER FROM ITS THERMAL BEHAVIOUR

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Abstract

This study deals with the determination of activation energy of a novel random copolyester (P1DID) based on 2,2'-oxydiethanol, 2,5-ditertiary butyl quinol with isophthaloyl chloride. Presence of ester group in the polymer enhances the reaction rate. The activation energy can be easily determined from the thermal degradation behaviour of polyester by three different methods (a) Murray and White (1955) (b) Coats and Redfern (1964) and (c) Doyle (1962). The activation energy determined by different methods are compared.

Keywords: Random copolyester, isophthaloyl Chloride, Thermal behaviour, Activation energy.

1. Introduction

Polyesters are high performance polymer with a range of properties including high thermal stability, resistance to chemical attack and lower dielectric constant. The synthesis of a novel random copolyester (P1DID) based on 2,2'-oxydiethanol, 2,5-ditertiary butyl quinol with isophthaloyl chloride as different diols and diacid chloride monomers done by solution polycondensation method is reported. The fundamental principles and techniques of thermal analysis have been discussed in detail (Slade and Jenkins 1996). Thermal analysis has become a widely accepted analytical tool and is very useful for rapid characterisation of many aspects of polyester structure and properties. TGA technique measures the mass of a substance as a function of temperature while the substance is subjected to a controlled temperature programme. TGA experiments were performed to elucidate the thermal behaviour and data generated that characterize the degradation. From a study of thermal degradation, the activation energy was calculated by three different methods.

2. Experimental

2.1 Materials and Methods

Chemicals of high purity were obtained from a variety of commercial sources: isophthaloyl chloride, 2,2'-oxydiethanol, 2,5-ditertiary butylquinol all purchased from Merk, Mumbai, India. Decontamination of solvent used

in this process was by 1,2-dichlorobenzene, kept overnight in anhydrous calcium chloride then filtered and purified using fractional distillation method at the temperature of 180°C and finally separated. Petroleum ether was dried in presence of anhydrous calcium chloride and finally distilled. Other solvents such as acetone, chloroform, carbontetra chloride, o-chlorophenol, dimethyl sulphoxide (DMSO) and methanol all in AR grade were used to ensure the solubility of the polymer.

2.2 Synthesis of Random Copolyester

Polymerization reaction, direct polycondensation of isophthaloyl chloride, 2,2'-oxydiethanol, 2,5-ditertiary butyl quinol in the ratio of 3:2:1 was taken in a reaction flask with 150ml of 1,2-dichloro benzene, refluxed at 130° to 160°C for 42hr in inert Nitrogen atmosphere with constant stirring. The content was cooled and extracted with petroleum ether and refrigerated overnight. Filtered crude sample was dissolved and evaporated in minimum amount of acetone to obtain pure polyester. The sample was dried in vacuum with phosphorous pentoxide, which produced a yield of 68 %.

3. Characterization

3.1 Thermogravimetric Analysis (TGA)

TGA trace follows relatively simple sigmoidal path. Thus, the sample weight decreases slowly as degradation begins, then decreases rapidly over a comparatively narrow temperature range and finally comes