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NOTE

Synthesis and Thermal Studies on Copolyesters of Succinoyl Chloride

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Co-polyesters of succinoyl chloride were synthesized by condensing two different diols with acid chlorides in *o*-dichloro benzene at 120-140ºC. Solubility was checked in some common organic solvents and they showed solubility in dimethyl formamide. The thermal analysis of copolyesters was carried out by TGA and DTA studies.

Key Words: Synthesis, Co-polyester, Succinoyl chloride, Thermal stability.

Polycondenzation of diols with dicarboxylic acids was often carried out in the melt in an inert solvent like xylene or *o*-dichloro benzene[1] Many random copolyesters have been synthesized by this method[2-4]. Copolyesters with substituted biphenylene moieties have been synthesized by Becker *et al.*[5] and Gaudiana *et al.*[6]. Kim and Hay[7] synthesized polyesters from biphenol monomers. The structure-property relationships of aromatic polyesters were explored by many research workers[8-10]. The thermal behaviour of copolyesters is known[11] to be very sensitive to their chemical structure and thermal stability. This paper describes the synthesis and thermal studies on copolyesters of succinoyl chloride from TGA and DTA thermograms. 1,2-Dichloro benzene and petroleum ether were dried over anhydrous calcium chloride and used.

Synthesis of poly[oxy(2,3-butylene)oxysuccinoyl-cooxy-(1,4-naphthyl)oxysuccinoyl] (PBNS): A 500 mL three necked round bottom flask equipped with a magnetic stirrer, nitrogen inlet, thermometer, reflux condenser with guard tube was charged with 2.7 mL of butane-2,3-diol (0.03 mol), 2.4026 g of 1,4-dihydroxy naphthalene (0.015 mol), 5 mL of succinoyl chloride (0.045 mol) and 150 mL of *o*-dichlorobenzene. The mixture was heated to reflux for 25-30 h at 130ºC in nitrogen atmosphere

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with constant stirring. After cooling the contents the mixture was poured in to about 500 mL of petroleum ether and allowed to stand for 24 h and filtered. The copolyester was washed with deionized water, dissolved in minimum amount of acetone and the clear solution was poured into water to get pure dull black co-polyester (yield 70-75 %).

Synthesis of poly[oxy(diethylene)oxysuccinoyl-cooxy-(1,4-naphthyl)oxysuccinoyl] (PDNS): In the same experimental set up as above a mixture of 2.9 mL of diethylene glycol (0.03 mol), 2.4025 g of 1,4-dihydroxy naphthalene (0.015 mol), 5 mL of succinoyl chloride (0.045 mol) and 150 mL of 1,2-dichloro benzene was taken and refluxed for 25-30 h at 130ºC. A similar procedure was adopted to get dry copolyester. The yield was around 50-55 %. Both the copolyesters were soluble in dimethyl formamide.

Thermal studies of copolyesters can be used to detect the phase transition on heating.The thermal stability of copolyesters is determined by thermo gravimetric analysis. The degradation of copolyesters was carried out in nitrogen atmosphere.In PBNS at 150ºC about 10 % of the copolyester was degraded. Between 150-590ºC about 70 % of the copolyester degraded. This is due to the scission of the copolyester. After 590ºC only 10 % degradation was observed. The TGA thermogram of PDNS shows that it is more stable than PBNS. The first 10 % degradation starts at 175ºC. 50 % degradation of PBNS requires 325ºC whereas PDNS require 362ºC. PDNS decomposes nearly 91 %. It is evident from the degradation temperature that the copolyester PBNS degraded at a lower temperature than PDNS. This may be due to the fact that the scission of the aliphatic chain in PBNS takes place at lower temperature than the polymer chainsin PDNS. The thermal stability of copolyesters PBNS and PDNS are given in Table-1.

TABLE-1
THERMAL STABILITY OF COPOLYESTERS

Copolyester	Temperature corresponding to Weight loss					
	10%	20%	30%	40%	50%	60%
PBNS	150	219	241	262	325	587
PDNS	175	237	268	293	362	385

Differential thermal analysis (DTA) of the copolyesters was carried out in nitrogen atmosphere using STA-409C instrument.The copolyesters showed mostly exotherms and few endotherms. The endotherms showed below 100ºC are small and may be readily assignable to glass transition temperature. Thermal transition of copolyesters such as glass transition temperature (T_g), mesophase formation (T_m), crystalline temperature (T_{cry}) decomposition temperature (T_d) are given in Table-2.

TABLE-2
THERMAL TRANSITION OF COPOLYESTERS

Copolyesters	Peak Temperature (ºC)			
	T_g	T_m	T_{cry}	T_d
PBNS	102.1	132.8	208	244.4
				305.1
PDNS	104	138	175	250.1
				320.8

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