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SYNTHESIS AND SPECTRAL ANALYSIS OF COPOLYESTERS

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ABSTRACT

Copolyesters are one of the most important class of polymers with repeating carboxylate group in their backbone. In the present work, copolyesters based on adipoyl chloride with aromatic diols were synthesized by solution polycondensation method. Vibrational band assignment has been made for some of the specific modes of vibration observed from FTIR spectra. The UV-Visible absorbance spectra of the polymeric material have been taken and the nature of chromophore present in the polymer chain is identified.

Key words : Copolyesters, FT-IR spectra, UV-Visible spectra.

INTRODUCTION

Copolyesters composed of aliphatic and aromatic units such as 1, 2-ethane diol, 1, 3-propane diol, 1, 4-butane diol, adipic acid and terephthalic acid combined in an appropriate ratio has proved to be biodegradable [1-3]. The biodegradation behaviour of copolyesters, which contain aliphatic and aromatic subunits, are dependent on composition, length and sequence distributions of aliphatic and aromatic units, crystallinity and melting temperatures[4,5]. Many random copolyesters have been synthesized by solution condensation of diols with dicarboxylic acids in a solvent like dichloro benzene[6-9]. In the present work, solution polycondensation method has been adopted for the synthesis of copolyesters viz., PRNA and PQNA.

EXPERIMENTAL

Synthesis of poly[oxy – (1, 3-phenylene) –oxy-adipoyl – co-oxy – (2, 3- naphthyl) – oxy-adipoyl] (PRNA)

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A 500 mL three necked round bottom flask equipped with a magnetic stirrer, nitrogen inlet, thermometer and reflux condenser with guard tube was charged with 1 mole of 2, 3-dihydroxy naphthalene, 2 moles of resorcinol, 3 moles of adipoyl chloride and 250 mL of 1, 2-dichlorobenzene. The mixture was heated to reflux for 25-30 hrs at 140-160 ^{0}C in nitrogen atmosphere with constant stirring and cooled to get pure shiny black copolyester. The yield was 58-60%.

Synthesis of poly [oxy– (1, 4-phenylene) –oxy-adipoyl – co-oxy –(2, 3 -naphthyl) – oxy-adipoyl] (PQNA)

In the same experimental set up as above, a mixture of 1 mole of 2, 3-dihydroxy naphthalene, 2 moles of quinol, 3 moles of adipoyl chloride and 250 mL of 1, 2-dichlorobenzene was heated to reflux for 25-30 hrs at 140-160 ^{0}C to get PQNA.

RESULTS AND DISCUSSION

FTIR Spectral analysis

The IR spectra of copolyesters were recorded using Perkin-Elmer spectrophotometer in the frequency region 4000-500 cm^{-1}. KBr pellet technique was employed for recording the IR spectra of copolyesters. The vibrational frequencies assigned for the copolyesters PRNA and PQNA are summarized in Tables 1 and 2.

Table 1 : IR Spectrum and vibrational band assignment of PRNA

Frequency (cm^{-1})		Vibrational band assignment
IR	Intensity	
3600	vw	Free –OH group
3076	m	Aromatic C-H asymmetric stretching
3030	m	Aromatic C-H symmetric stretching
2963	m	Aliphatic C-H asymmetric stretching
2879	m	Aliphatic C-H symmetric stretching
1694	vs	>C=O stretching of ester group
1626	vw	C=C stretching vibration of aromatic homocyclic compound
1509	vw	Aromatic C=C stretching

vs- very strong, vw – very week, s – strong, w – week, m-medium

Table 2 : IR Spectrum and vibrational band assignment of PQNA

Frequency (cm^{-1})		Vibrational band assignment
IR	**Intensity**	
3600	vw	Free –OH group
3071	m	Aromatic C-H asymmetric stretching
3029	w	Aromatic C-H symmetric stretching
2934	m	Aliphatic C-H asymmetric stretching
2875	w	Aliphatic C-H symmetric stretching
1751	vs	>C=O stretching of ester group
1698	s	>C=O stretching of ester group
1602	w	Aromatic C=C stretching
vs- very strong, vw – very week, s – strong, w – week, m-medium		

UV-Visible spectral analysis

The UV-Visible absorbance spectra of the copolyesters have been recorded using Shimadzu UV160A. The copolyesters PRNA and PQNA, contain the substituted naphthalene as chromophore and have absorption maximum around 340 -450 nm. It may be evident that the value corresponding to the wavelength maxima is not influenced by length of the spacer in polymer chain but influenced by the nature of condensed rings present in them.

A necessary preliminary assignment of bands in the infrared spectra gives us a thorough knowledge of the number and kinds of normal modes of vibration and their structure[10]. Hence in the present investigation, a detailed work is done on the analysis of vibrational bands to confirm the structure of repeating units of the synthesized copolyesters.

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