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SYNTHESIS OF POLYESTER CONTAINING CHALCONE LINKAGE AS CONDUCTING POLYMER

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ABSTRACT :

Synthesis and characterization of polyesters comprising 4,4'-dihydroxy bischalcone as main chain moiety is reported here. Polyester chalcones were obtained by condensation of 4,4'-dihydroxy chalcones with adipic and terepthalic acid respectively. The polymers were characterized by IR and elemental analysis. The thermal and electrical behaviour were studied by TGA, DSC, and conductivity techniques respectively. The polymers were found to behave like semi-conductors and were thermally stable.

INTRODUCTION :

Although polyesters are not usually considered to be in a category of thermally stable materials. Some significant advances have been made toward this end (1-6). Polyester prepared by the incorporation of chalcone group into main chain may enable them to be used as heat resistant materials as well as semiconductors. The use of chalcone in polyester formation has been explored here utilizing 4,4'-dihydroxy bischalcone.

The present paper reports the synthesis of polyester chalcone by condensing dihydroxy chalcone with adipic acid as well as terephthalic acid. The polymers were characterized by elemental IR and thermal analysis. The electrical behaviour of polymers was also studied.

Material :

p-Hydroxy acetophenone, terephthaldehyde, adipic acid and terephthalic acid of were used for the preperation of polyester chalcone.

Synthesis :

(i) Preparation of 4,4'-dihydroxy bischalcone :

In a 250 cc. conical flask, p-hydroxy acetophenone (0.02 mole) and terephthaldehyde (0.01 mole) in methanol were taken and 40% methanolic solution of KOH was added dropwise with stirring. The stirring was continued for 4-5 hrs. The solution was kept overnight and then decomposed by 1:1 dilute hydrochloric acid. The precipitate which separated out, were filtered and washed with cold water and dried. The product was crystallized from 50:50 ethonol acetic acid mixture and finally with acetic acid. m.p. 310°C.

(2) Preparation of Acid chloride :

Terephthanoyl and adipinoyl chlorides were prepared by reacting corresponding terephthalic acid and adipic acid with thionyl chloride.

(3) Preparation of polyester chalcone by condensation polymerization :

4,4'-dihydroxy bischalcone (0.01 mole) was dissolved in 10 ml of dry pyridine. Acid chloride

was also dissolved in 10 ml of dry pyridine. Both were mixed with a constant stirring in an ice bath. The reaction was kept overnight and then acidified with 1:1 dilute hydrochloric acid. Unreacted monomer was removed by giving alkali wash. Compounds were purified by refluxing in alcohol/acetic acid. The polyester chalcones based on adipic acid and terephthalic acid are designated as PECA & PECT respectively.

Characterization :

I.R. spectra of chalcone and its polymers viz.; PECA and PECT were recorded on schimadzu IR-408 with KBr pellets.

Thermogravimeteric (TGA) and differential calorimetry (DSC) analysis of polymers were performed with Schimadzu thermal analyser-30 in air at a heating rate of $10^{o}C$/min.

Electrical conductivity of both polymers PECA and PECT was measured by systronic make De voltmeter. The current measurement was done by W.G. pye make ballistic galvanometer of sensitivity 0 05 amp/min. The polymer samples were made into

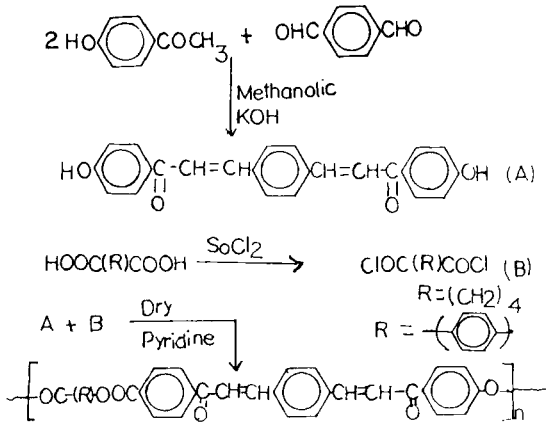
a pellet, placed in temperature controlled chamber and heated to the required temperature.

Results and Discussions :

Synthetic route for the polyester chalcone is illustrated in scheme (I).

1. **IR Spectral Analysis :**

IR spectra of 4,4'-dihydroxy bischalcone, PECA and PECT are shown in Fig. 1(a). It showed the characteristic -OH stretching frequency at 3600-3000 cm^{-1}. The -C=O stretching is observed at 1640 cm^{-1} and is lowered due to its conjugation with C=C and aromatic ring. The =CH stretching frequency is observed at 970 cm^{-1}. The other characteristic absorption bands are 1600, 1350, 1290, 1170, 907, 810 cm^{-1}. IR spectrum of PECT (Fig 1(b)) shows the characteristic peak at 1740 cm^{-1} due to ester linkage attached to aromatic group. The ester linkage observed in IR spectrum of PECA Fig. 1(c) at 1680 cm^{-1} is due to aliphatic chain attached to ester linkage. The other characteristic bands observed in polyesters are similar to that of 4,4'-dihydroxy chalcone.



2. Conductivity :

The effect of temperature on conductivity is studied by measuring the conductivity at different temperatures by heating the sample in a closed chamber. The temperature dependence of electrical conductivity can be described by using the equation,

$$\sigma = A \exp\left(\frac{-W}{KT^{1/3}}\right)$$

Where, σ = The conductance

W = Activation energy of conductance

A = Constant

T = Temperature

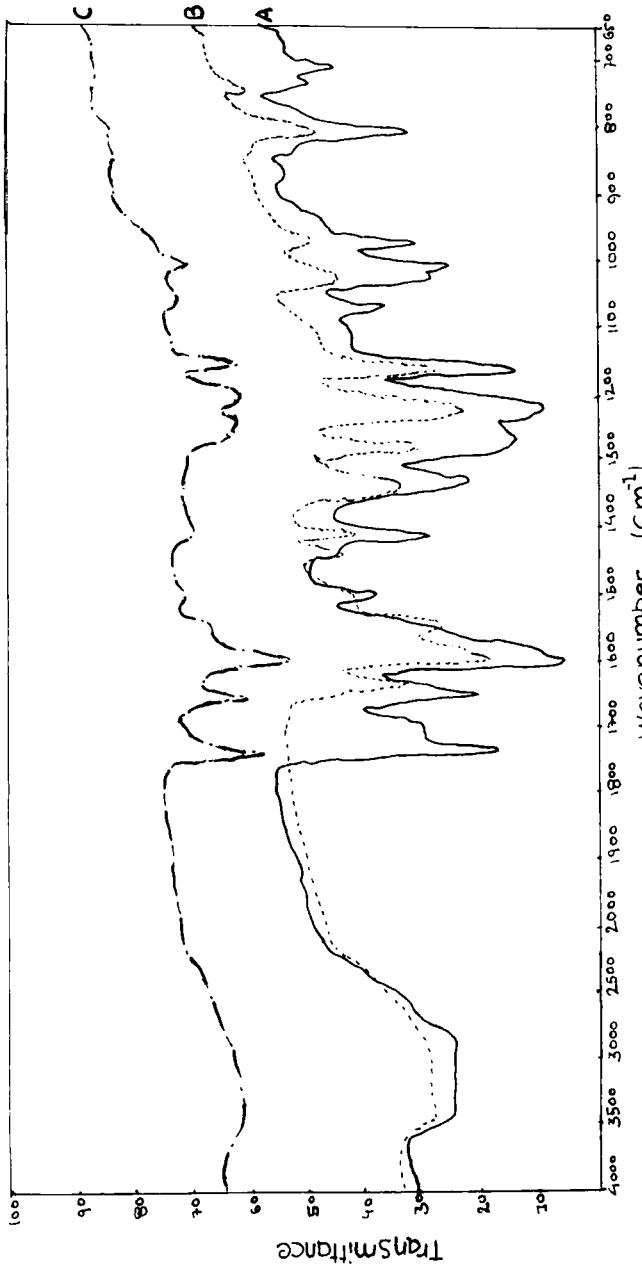


Figure 1

The measured values of conductivity are plotted semilog arithmetically in fig. 2, as a function of $1/T^{1/3}$. The increase in conductivity with increase in temperature shows that these polymers behave like semiconductors. Moreover as the plot of log σ versus $1/T^{1/3}$ are straight line, it shows that the mechanism of conduction may be due to variable range hopping.[7] Energy of activation data of conductance (W) calculated for PECA and PECT are shown in Table 1. The Values are comparable to modified Poly (acetylenes)[8].

3. Thermal Characterization :

The thermograms (TGA) of PECA and PECT at heating rate $10^{o}C$/min in air are shown in fig. 3. From thermograms it is clear that the weight loss is rapid in the temperature range 400-600^{o}C and slow in the temperature range 100 to 400^{o}C. Thus the degradation involves two steps. Table 2, gives the temperature at which 10%, 30% and 50% decomposition take place.

The values of activation energy (E) determined according to Broido method[9] are listed

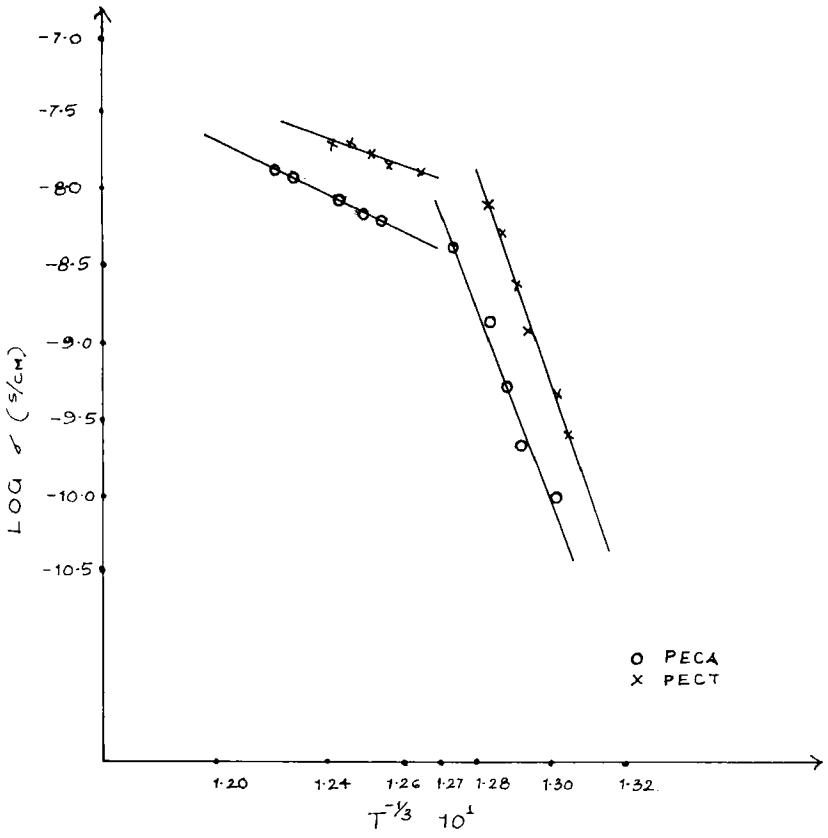


Figure 2

TABLE : 1

Energy of activation (W) of Conductance of Polyester 4,4'-bischalcone.

Polymer	Activation Energy (ev)	
	Ist region W	IInd region W
PECT	0.13	0.033
PECA	0.14	0.016

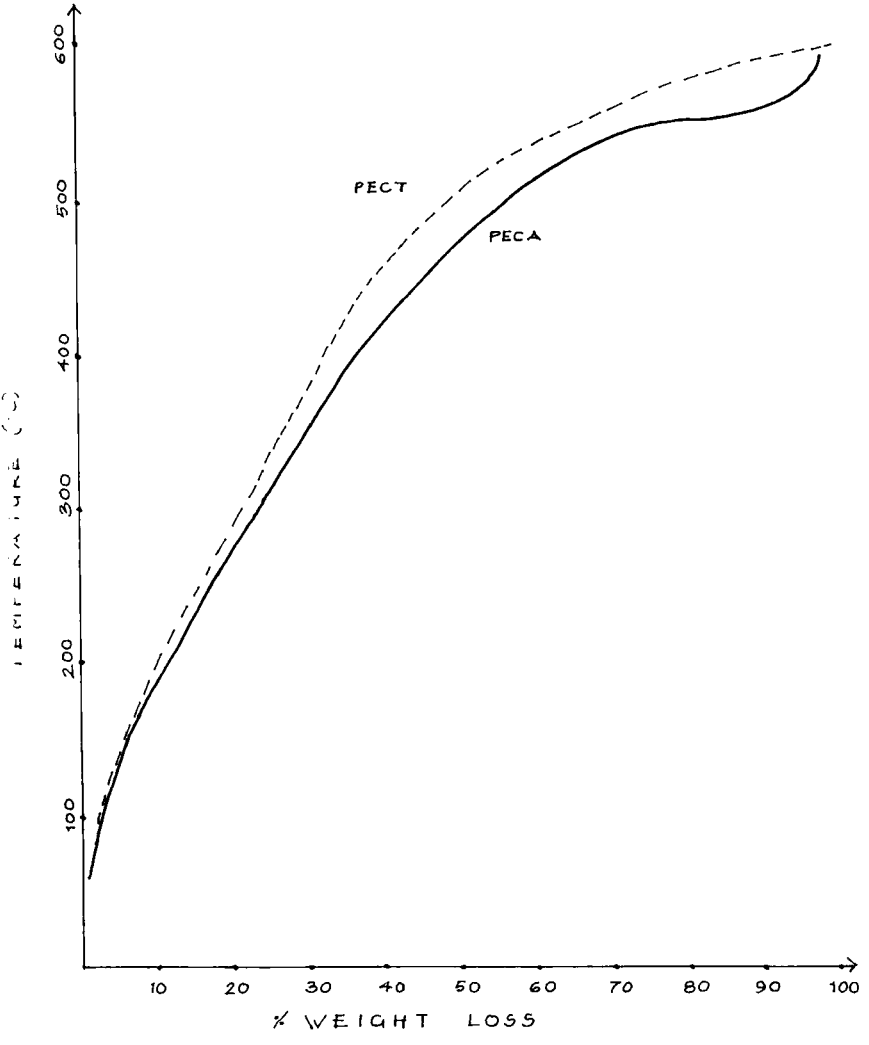


Figure 3

TABLE : 2

Thermal Characteristics of Polyester 4,4'-bischalcone :

Polymer	% decomposition			Ea (kJ/mole)		T_g °C
	10	30	50	I	II	
PECA	175	315	475	15.1	77.6	230
PECT	200	375	515	13.0	98.8	-

in Table 1. The value of E of step II is quite higher than in step I in both the polymers. This indicates that the product obtained at the end of first step degradation has better thermal stability. Further the value of E of step II for polymer PECT is considerably higher to that of PECA. This may be due to the presence of aromatic ring of terephthalic acid in PECT, linked with chalcone linkage to that of PECA having an aliphatic chain. These polymers were also subjected to DSC analysis and were found to have high Tg. Polyester chalcones PECA and PECT were found to be insoluble in almost all the solvents.

Conclusion :

The synthesized polyesters containing chalcone have good thermal stability and behave like semi-conductors.

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