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Synthesis and Characterization of New Unsaturated Polyesters Containing 4-Phenylcyclohexanone Moiety in the Main Chain

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Two series of new unsaturated polyesters were prepared from 2,6-bis(*p*-hydroxidebenzylidene)-4-phenylcyclohexanone (I) and 2,6-divanillyidene-4-phenylcyclohexanone (II) with adipoyl, isophthaloyl, sebacoyl and terephthaloyl dichlorides utilizing the interfacial polycondensation technique at ambient temperature. In addition to that, the model compounds were synthesized by reacting (I) and (II) with benzoyl chloride. The model compound and polyester samples have been characterized by elemental and spectral analyses. The unsaturated polyesters have inherent viscosities of 0.96–1.63 dl/g. All the polyesters are amorphous and most of them are partially soluble in most common organic solvents, but easily soluble in concentrated sulfuric acid. Their glass transition temperatures (T_g) range from190.15 to 245.28°C, and the temperatures of 10% weight loss as high as 180 to 220°C in air, indicating that these aromatic polyesters have high T_g and excellent thermal stability.

Keywords: unsaturated; polyesters; dibenzylidenecycloalkanones; 4-phenylcyclohexanone; synthesis; characterization

1 Introduction

Unsaturated polyesters are one of the most important matrix resins for composite applications. They possess a wide range of properties and are used in marine, automobile, construction, sport, self-extinguishing materials (1–4) and they have also led to the need and development of high performance unsaturated polyesters. Several researchers have reported the preparation of unsaturated polyesters by the polycondensation of dicarboxylic acids (or acid anhydrides) with diols, where at least one of the monomers contains an unsaturated carbon-carbon bond (C=C) (5–8).

Unsaturated polyesters are generally difficult to process because of their limited solubility in organic solvents and their high melting temperature or high glass-transition temperature by virtue of their rigid structures. Therefore, development of polyesters for use at high temperature with improved solubility is an important goal. One of the approaches to improve solubility of the polymers without extreme loss of their high thermal stability is the introduction of polar and flexible groups into the polymer backbone (9, 10). The incorporation of bulky pendent groups can also provide beneficial effect for solubility because this approach produces a separation of chains and lowering the chain packing with a gain of free volumes. Bulky side groups also decrease the molecular mobility, so that the overall observable effect is an increase of the glass transition temperature and an improvement of solubility at the same time (11, 12).

We now outline the synthesis and characterization of new unsaturated polyesters containing 4-phenylcyclohexanone moiety in the main chain. A major target of this work was to study the effect of phenyl ring, as a rigid moiety, upon the properties of unsaturated polyesters, including their thermal stability, solubility and crystallinity.

2 Experimental

2.1 Instrumentation

Melting points were determined on a Perkin-Elmer 240°C electro thermal melting point apparatus and are uncorrected. Infrared spectra were recorded on a Shimadzu 2110 PC spectrophotometer with KBr pellets. The ^{1}H-NMR spectra were recorded on a GNM-LA 400 MH_z NMR spectrophotometer at room temperature in DMSO using TMS as the internal reference. Viscosity measurements were made with a 0.5% (w/v) solution of polymers in DMSO at 25°C using an Ubbelohde suspended level viscometer. TGA and DTG measurements were performed on V 5.1 A DuPont 2000 thermal

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analyzer at a heating rate 10°C/min in air. DSC was carried out in a nitrogen atmosphere using a Shimadzu DSC-50 thermal analyzer. The X-Ray diffractograms of the polymers were obtained with a Phillips X-ray unit (Phillips generator Pw-1710) and Ni-Filtered $CuK_\alpha\alpha$ radiations.

2.2 Reagents and Solvents

4-Hydroxybenzaldehyde(BDH), 4-hydroxy–3- methoxybenzaldehyde (Merck) and 4-Phenylcyclohexanone (Aldrich) were used without purification. Terephthaloyl chloride (Aldrich) was recrystallized from n-hexane (mp 83–84°C) and isophthaloyl chloride (BDH) was recrystallized from n-hexane (mp 44°C). Adipoyl and sebacoyl dichlorides (Merck) (13) were freshly distilled. Adipoyl dichloride at 125°C/11 Torr, and sebacoyl dichloride at 182°C/16 Torr were used. Benzoyl chloride (BDH) and sodium hydroxide were of analytical grade. Dichloromethane and all other solvents were of high purity and further purified by standard methods (14, 15).

2.3 Monomer Synthesis

2.3.1 2,6-Bis(p-hydroxybenzylidene)-4-phenylcyclohexanone (I)

A mixture of 0.2 mol *p*-hydroxybenzyldehyde and 0.1 mol 4-phenylcyclohexanone was warmed in the absence of solvent on a water-bath for 2 h. The warmed reaction mixture was treated with 50% absolute ethanol, whereby a highly yellowish solid precipitated. The solid material was filtered off, dried and recrystallized twice from ethanol. Yellow needles were obtained, yield 85%, m.p. 132°C.

Calculated composition of $C_{26}H_{22}O_3$: C, 81.67; H, 5.75%. Found: C, 81.21; H, 5.39%. IR (KBr): at 1595 cm^{-1} (s, C=C), at 1690 cm^{-1} (s, C=O), at 2850–2940 (C-H of cyclohexanone), and at 3490–3500 cm^{-1} (br, OH stretching). ^{1}H-NMR (DMSO-d_6): at 9.10 (s, 2H of 2OH), at 8.00–7.00 (m, 13H of Ar-H), at 5.90 (s, 2H of 2CH=C), at 2.95 (m, 4H of $2CH_2$ cyclohexanone), and at 1.25 (middle, H of CH cyclohexanone) ppm (Figure 1).

2.3.2 2,6-Divanillyidene-4-phenylcyclohexanone (II)

A mixture of 0.2 mol *p*-hydroxy-3-methoxybenzyldehyde and 0.1 mol 4-phenylcyclohexanone was warmed in the absence of solvent on a water-bath for 2 h. the warmed reaction mixture was treated with 50% absolute ethanol, whereby a highly yellowish solid precipitated. The solid material was filtered off, dried and recrystallized twice from ethanol as yellow crystalline needles. Yield 90%, m.p. 165°C.

Calculated composition of $C_{28}H_{26}O_5$: C, 76.01; H, 5.88%. Found: C, 76.18; H, 5.32%. IR (KBr): at 1595 cm^{-1} (s, C=C), at 1660 cm^{-1} (s, C=O), at 2850–2940 (C-H of cyclohexanone) and at 3490–3500 cm^{-1} (br, OH stretching). ^{1}H-NMR (DMSO-d_6): at 9.10 (s, 2H of 2OH), at 7.80–7.10 (m, 11H of Ar-H of dibenzylidene), at 5.60 (2H of 2CH=C), at 4.20 (s, 6H, 2-OCH_3), at 2.50–2.95 (m, 4H of $2CH_2$ cyclohexanone), and at 1.30 (middle, H of CH cyclohexanone) ppm.

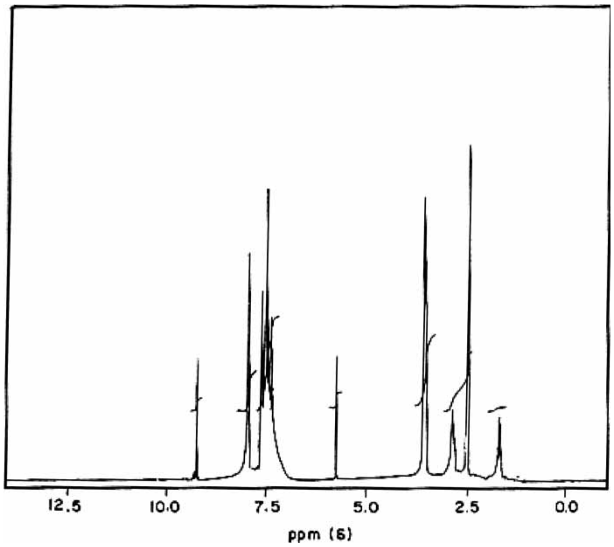


Fig. 1. ^{1}H-NMR spectrum of monomer I.

2.4 Synthesis of Model Compounds A and B

2.4.1 General Procedure

2,6-Bis(*p*-hydroxybenzylidene)-4-phenylcyclohexanone I or 2,6-divanilly-ylidene-4-phenylcyclohexanone II (6 mmol) was dissolved in 20 ml sodium hydroxide solution (12 mmol) and stirred at 25°C. After stirring, benzoyl chloride (12 mmol) was carefully added within 20 min. At the end of the reaction time, a highly yellowish solid precipitated. The solid product was filtered off, washed with water, dried and recrystallized from an appropriate solvent.

2.4.2 2,6-Bis(p-hydroxybenzylidene)-4-phenylcyclohexanone Dibenzoate (A)

An analytical sample was obtained by recrystallization from ethanol, m.p. 168°C yield 78%. Calculated composition of $C_{40}H_{30}O_5$: C, 81.35; H, 5.08%. Found: C, 81.31; H, 5.26%. IR (KBr): at 1595 cm^{-1} (s, C=C), at 1735 cm^{-1} (s, C=O of ester), at 1680 cm^{-1} (m, C=O of cyclohexanone). ^{1}H-NMR (DMSO-d_6): at 8.00–7.80 (m, 8H, Ar-H of dibenzylidene), at 7.80–7.50 (m, 5H, Ar-H of phenylcyclohexanone), at 7.50–7.20 (m, 10H, Ar-H of dibenzoate), at 6.20 (s, 2H, 2CH=C), at 3.20 (s, 4H of $2CH_2$ cyclohexanone), and at 1.55 (middle, H of CH cyclohexanone) ppm (Figure 2).

2.4.3 2,6-Divanillyidene-4-phenylcyclohexanone Dibenzoate (B)

A pure sample was obtained by recrystallization from ethanol, m.p. 142°C, yield 72%. Calculated composition of $C_{42}H_{34}O_7$: C, 77.53; H, 5.23%. Found: C, 77.34; H, 5.37% IR (KBr): at 1735 cm^{-1} (s, C=O of ester) 1670 cm^{-1} (m, C=O of cyclohexanone), and at 1590 cm^{-1} (s, C=C). ^{1}H-NMR (DMSO-

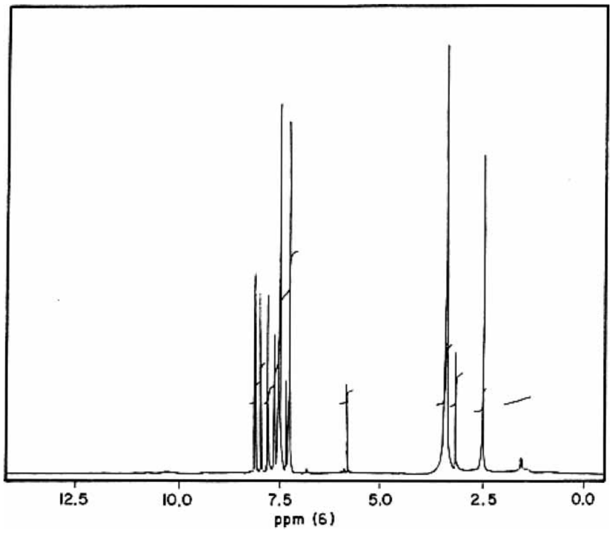


Fig. 2. ^{1}H-NMR spectrum of model compound (A).

d$_6$): at 7.90–7.75 (m, 6H, Ar-H of dibenzylidene), at 7.75–7.50 (m, 5H, Ar-H of phenylcyclohexanone), at 7.50–7.30 (m, 10H, Ar-H of dibenzoate), at 5.90 (s, 2H, of 2CH=C), at 4.2 (s, 6H, of 2OCH$_3$), at 2.75–2.60 (m, 4H of 2CH$_2$ cyclohexanone), and at 1.40 (middle, H of CH cyclohexanone) ppm.

2.5 *Polymer Synthesis*

In a three-necked flask equipped with a mechanical stirrer (2000/rpm/min), dry nitrogen inlet and outlet, and dropper, a mixture of 1 mmol dibenzylidene 4-phenylcyclohexanone (I or II) 25 ml methylene chloride, and sodium hydroxide solution (2 mmol) was introduced. After mixing, 1 mmol of acid chlorides (III$_{a-d}$) dissolved in 30 ml methylene chloride was added over a period of 2 min at 25°C and vigorously stirred. After complete addition of acid chloride, stirring was continued for 1–2 h, and a highly yellowish solid polymer separated out. The solid polymer was filtered off, washed with water, hot alcohol, and dried under reduced pressure (1 mmHg) at 80°C for 1 day.

The synthesized polyesters, their yield and some of their properties are listed in Table 1.

3 Results and Discussion

3.1 *Monomers*

The preparation of 2,6-bis(*p*-hydroxybenzylidene)-4-phenylcyclohexanone (I) and 2,6-divanillyidene-4-phenylcyclohexanone (II) used in this study was accomplished by using the condensation reaction of *p*-hydroxybenzaldehyde or vanilline with 4-phenylcyclohexanone in the absence of solvent, respectively. The structure of these new monomers was elucidated by elemental and spectral analyses (see Experimental). The preparation of I and II is shown in Scheme 1.

3.2 *Model Compounds*

Before attempting the polymerization, model compounds (A) and (B) were prepared by the reaction of 2,6-bis(*p*-hydroxybenzylidene)-4-phenylcyclohexanone(I) and 2,6-divanillyidene-4-phenylcyclohexanone (II), respectively, and benzyl chloride in sodium hydroxide solution. Based on good agreement between calculated and found analytical data, IR and NMR, these reactions are depicted in (Scheme 2).

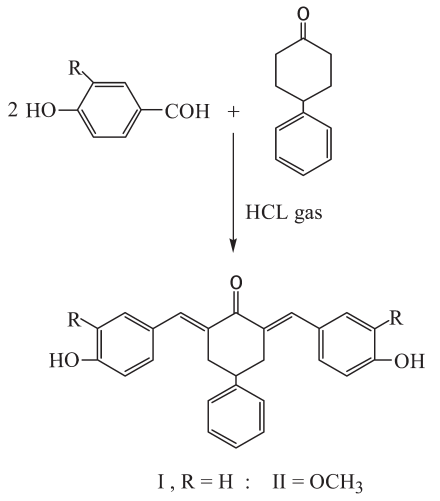
3.3 *Polymerization*

The polymerization of 2,6-bis(*p*-hydroxybenzylidene)-4-phenylcyclohexanone (I) or 2,6-divanillyidene-4-phenylcyclohexanone (II) with adipoyl, sebacoyl, isophthaloyl, or terephathaloyl dichlorides, results in the formation of novel unsaturated polyesters. Typical polymerization conditions involved the reaction of the stoichiometric amount of (I) or (II) with diacid chlorides (III$_{a-d}$) and the application of interfacial polycondensation with NaOH/CH$_2$Cl$_2$ at ambient temperature afforded new unsaturated polyesters as shown

Table 1. Results of elemental analyses, and some physical properties of polymers IV$_{a-d}$ and V$_{a-d}$

Polymer number	Analysis C% Calc.	C% Found	H% Calc.	H% Found	Yield (%)	$\eta_{inh}{}^a$ (dl/g)	Appearance of the polymer
IV$_a$	79.68	79.47	4.68	4.18	83	1.34	Brittle film
IV$_b$	79.68	78.91	4.68	3.96	85	1.47	Brittle film
IV$_c$	78.04	77.61	5.69	4.94	80	1.06	Yellow powder
IV$_d$	78.83	78.78	6.56	6.16	79	0.99	Yellow powder
V$_a$	75.52	75.51	4.89	4.03	80	1.24	Brittle film
V$_b$	75.52	75.19	4.89	4.21	88	1.63	Brittle film
V$_c$	73.91	73.89	5.79	4.98	81	1.22	Yellow powder
V$_d$	75.01	74.51	6.57	6.09	78	0.96	Yellow powder

[a] Inherent viscosity measured in DMSO at 25°C.

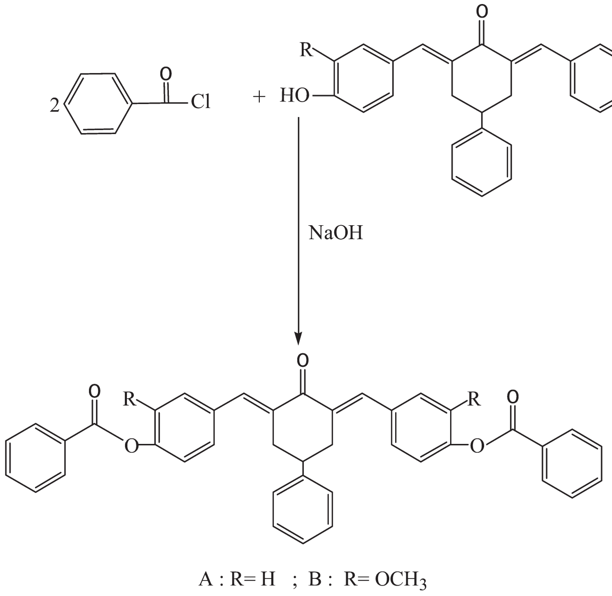


Sch. 1. Synthesis of Monomers I and II.

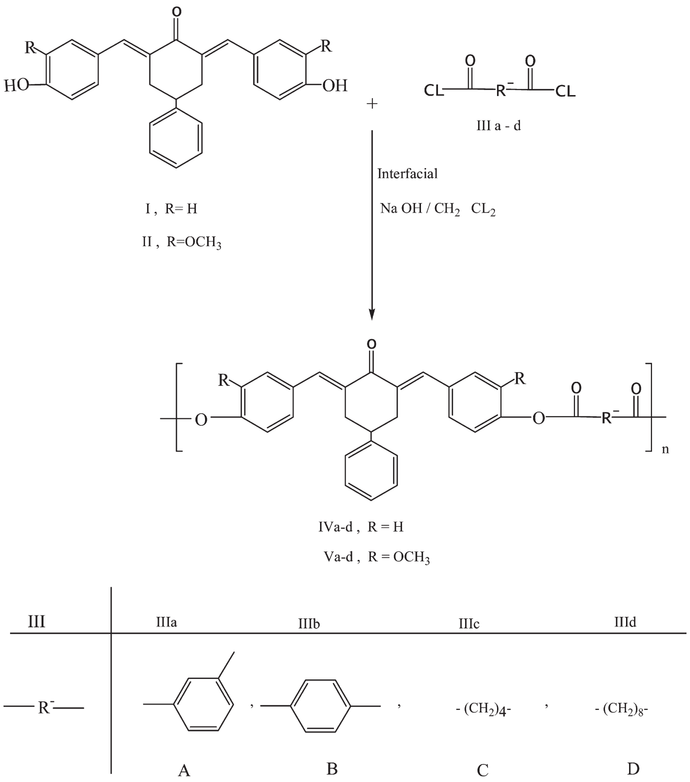
in (Scheme 3). Reaction times varied from 1 to 2 h. The polymerization mixtures gave a highly yellowish solid. Polymers were immediately isolated (see experimental) at the end of the reaction period.

Elemental analyses were performed on all new monomers and model compounds, as well as on all of the polymers. While the analysis for the model compounds are within 0.19% of the theoretical values, the analyses for the polymers (Table 1) deactivated from 0.30 about 0.77% of the theoretical values. However, it is not uncommon for polymers to trap solvent molecules within the polymer matrix, especially polymers of high molecular weight and those polymers containing polar groups which are capable of hydrogen bonding with solvent molecules.

Spectral data support the structural assignment for the polymers and are in agreement with spectral data obtained for the model compounds. IR data obtained in KBr disks for all polyesters indicated the disappearance of the characteristic absorption band of the OH group and the appearance of the C=O of ester groups at 1735–1745 cm^{-1}, at 1690–1700 cm^{-1} for the C=O of 4-phenylcyclohexanone, at 1600–1640 cm^{-1} for C=C groups, at 2985 cm^{-1} for C-H aliphatic, and the other characteristic absorption bands for the rest of the molecule (Figure 3).



Sch. 2. Synthesis of model compounds A and B.



Sch. 3. Synthesis of polyesters IV_{a-d} and V_{a-d}.

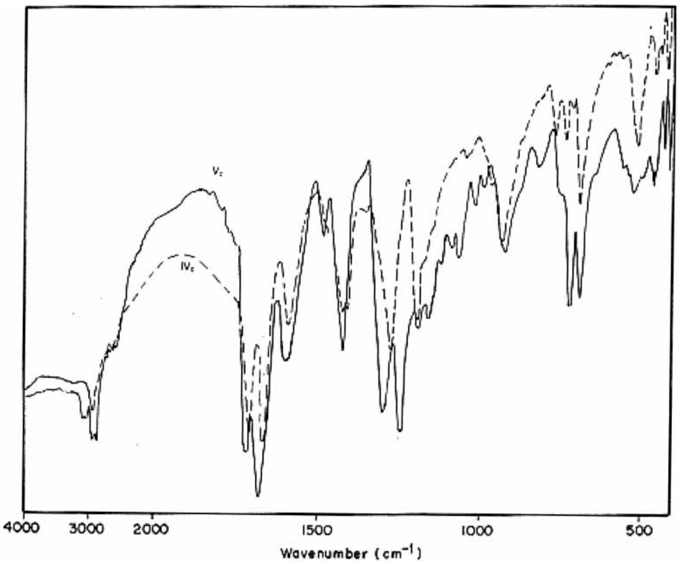


Fig. 3. IR spectra of polymers IV_c (---) and V_c (- - -).

The inherent viscosities (η_{inh}) of polyesters IV_{a-d} and V_{a-d} were determined in DMSO at 25°C with an Ubbelohde suspended level viscometer. The inherent viscosity value is defined as:

$$\eta_{inh} = (2.3\ \log t/t_O)/C$$

where t_o and t represent the viscometer flow periods for DMSO and the polymer solution, respectively; while solution concentration C is 0.5 g/100 ml. It can be clarified from Table 1 that polyesters having high inherent viscosity values and this may be attributed to high molecular weight of these polymers with inherent viscosities ranged from 0.96–1.63 dI/g.

The solubility of unsaturated polyesters synthesized in this study was determined for the powdery samples in various organic solvents including dimethylsulfoxide (DMSO), N-N dimethylformamide (DMF), N-methylpyrrolidone (NMP), tetrahydrofuran (THF), Dichloromethane (DMC), Tetrachloromethane (TCE), CHCl3-acetone (1:1) and concentrated sulphuric acid at 3.0% (w/v) and the results are listed in Table 2. It was found that all the polymers dissolved readily in concentrated H_2SO_4 at room temperature giving a deep red color due to the instability of the polymers. All polymers were soluble in various aprotic polar solvents such as DMSO, DMF, NMP, and THF. The good solubility of these polymers may be elucidated by loose packing of the macromolecules because of pendent phenyl rings and low crystallinity (16). In common organic solvents and halogenated hydrocarbons, the polymers $IV_{a,b,c}$ and $V_{a,b}$ were partly soluble (except polymer IV_d, and $V_{c,d}$ are soluble) which may be attributed to the presence of four or eight methylene groups, with the rigid benzene position of the main chain causing an increase in solubility. It should be noted that the polymer V_c is more soluble than IV_c, and this behavior could be attributed to the steric hindrance effect of the methyoxyl group, which causes both unsymmetrical orientation thereby making salvation easier (17). Therefore, the bulkiness of the pendent groups may play a more important role than rigidity in the improvement of the polyesters solubility (18).

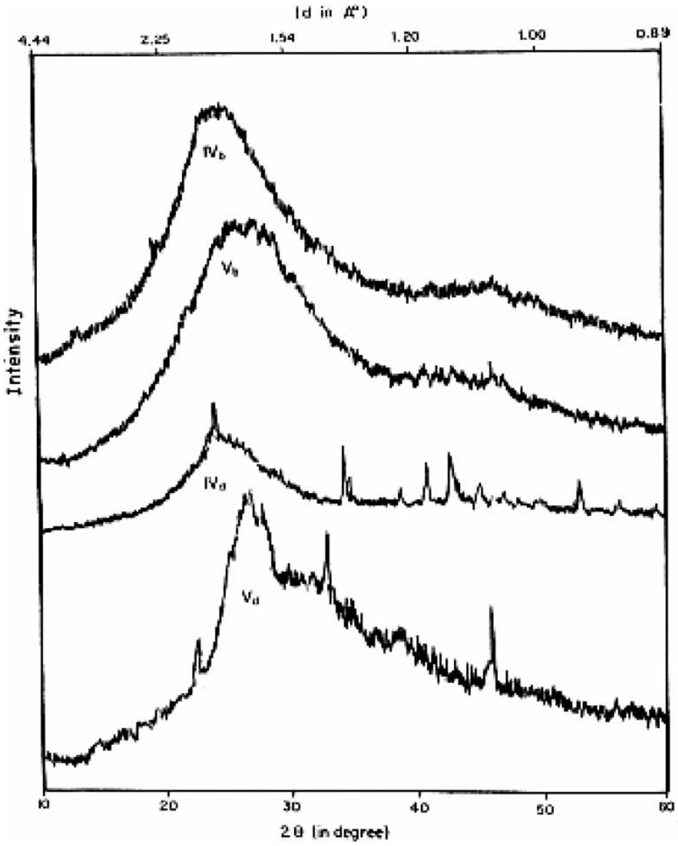


Fig. 4. X-ray diffraction patterns of polymers $IV_{b,d}$ and $V_{b,d}$.

The X-ray diffraction confirmed that the polymers were semi-crystalline because of a small portion of crystalline peaks in the region $2\theta = 10–60°$ or (d in A°) as shown in (Figure 4). In spite of a small crystalline region in every case, amorphous patterns are due to a lower packing of the unsymmetrical polymer back bones. In particular, the bulky phenyl substituent along the backbone most probably is the main reason for the amorphous nature of the present

Table 2. Solubility characteristics of polymers IV_{a-d} and V_{a-d}

Polymer number	DMF	DMSO	NMP	THF	DMC	Chloroform + Acetone (1:1)	TCE	Concentrated H_2SO_4
IV_a	+	++	+	+	+	+	−	++
IV_b	++	++	++	+	+	+	−	++
IV_c	++	++	++	++	+	+	+	++
IV_d	++	++	++	++	++	++	+	++
V_a	+	++	++	+	+	+	−	++
V_b	++	++	+	+	+	+	+	++
V_c	++	++	++	++	++	++	+	++
V_d	++	++	++	++	++	++	++	++

(++) Soluble at room temperature (RT).
(+) Partially soluble at (RT).
(−) Insoluble at (RT).

Table 3. Thermal properties of polymers IV_{a-d} and V_{a-d}

Polymer number	(Temperature (°C) for various % decomposition)[a] 10	20	30	40	50	Thermal behavior of polyesters T_g(°C)[b]	Tm (°C)[c]
IV_a	215	260	330	395	475	210.13	—
IV_b	220	270	350	410	490	245.28	406.19
IV_c	190	240	295	358	400	205.22	402.82
IV_d	185	235	295	340	355	198.71	—
V_a	203	245	325	390	455	205.32	407.36
V_b	210	255	310	385	470	219.73	408.66
V_c	185	240	290	355	385	201.65	—
V_d	180	225	285	325	340	190.15	385.45

[a] The values were determined by TGA at a heating rate of 10°C/min.
[b] Measured by DSC at a heating rate of 10°C/min in nitrogen.
[c] Measured by DSC at a heating rate of 10°C/min in nitrogen.

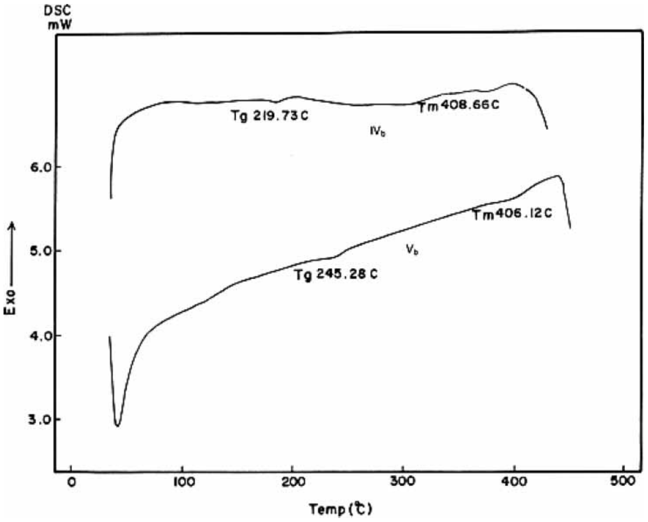


Fig. 5. The DSC curves of polymers IV_b and V_b.

polyesters, it is also evident that the presence of the phenyl substituent in the polymer main chain tends to destroy the polymers ability to form crystalline structures. The presence of eight methylene groups in polymers IV_d and V_d increases polymer chain flexibility, and that might be responsible for the approach and mutual attractions of adjacent chains and thus, induce small crystalline peaks. Moreover, the presence of C=O as polar groups in addition to high C=C bond levels induces some order between two adjacent chains in the polymer, leading to some extent of crystallinity (17). It should be noted that the inclusion of 4-phenylcyclohexanone moiety as a pendent group in the polymer main chain causes a slight decrease in the degree of crystallinity compared with those polyesters based on diarylidenecycloalkanones which were revealed in the literature (19, 20).

DSC and TGA measurements were performed to study the thermal behavior and thermal stability of the prepared unsaturated polyesters. The results are collected in Table 3. The investigation of DSC curves revealed that T_g of polyesters as a second order transition were in the range of 190.15–245.28°C. Figure 5 shows the polyesters IV_b and V_b derived from diols with bulky pendant groups and aromatic diacid chlorides, and showed higher T_g values. As expected, and shown in the Table 3, the polyester V_b, which was prepared from symmetrical diol I with pendant group and diacid chlorides III_b has the highest T_g. This is reasonable because a bulky pendent group inhibits the free rotation of the polymer chain and leads to an enhanced T_g value (21). Polymer VI_d, which was prepared from diol II containing methoxy group with pendant group and diacid chlorides III_d has the lowest T_g. This decrease in the glass transition temperature can be explained by some compatibilization of flexible methylene units with rigid blocks. The DSC curves also showed endothermic peaks of polyesters which appeared at around 385.45–408.66°C, interpreted as a melting process accompanied by decomposition.

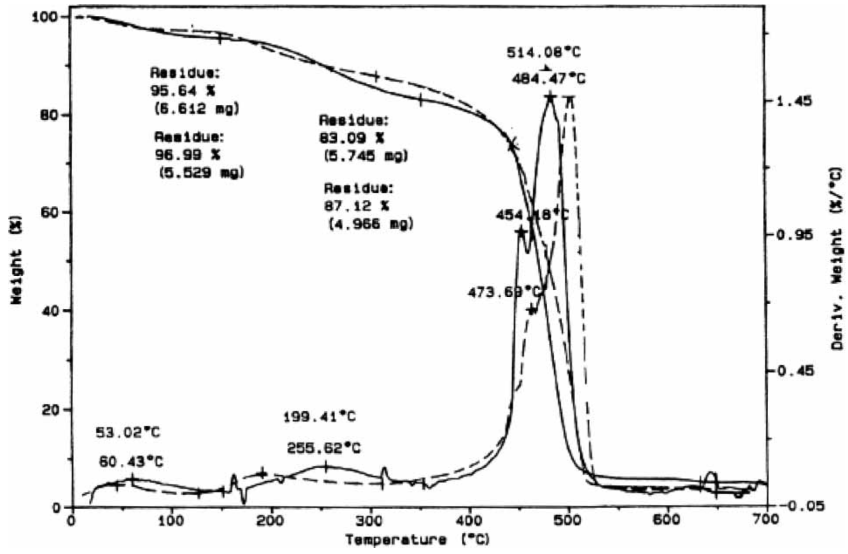


Fig. 6. The TGA and DTG traces of polymers IV_b (- - -) and V_b (—).

The thermal behavior of these polyesters, IV_{a-d} and V_{a-d}, was evaluated by TGA in air at a heating rate 10°C min^{-1}. In Figure 6 the TGA curves show a small weight loss in the range of 2–6% starting at 130°C until 160°C, which may be attributed to loss of adsorbed moisture and entrapped solvents, respectively. The thermographs also indicated in Figure 6 that the polymers decompose in two stages and the mass loss is seen to be rapid between ~190–473°C for the polymer IV_b in the first region, and between 473–514°C in the second region. For the polymer V_b, the mass loss was rapid between ~185–454°C in the first region and between ~454–484°C in the second region. All the studied polymers decompose in the first stage between 175 and 473°C, whereas in the second stage, they decompose from 380 to 514°C, which depends on the nature of the polyester. The expected nature of decomposition of these polymers is a pyrolytic oxidation of -C═C- (22), scission of many bonds, in addition to the formation of char as an end product. The temperature for 10% weight loss is considered to the polymer decomposition temperature, and it ranges between 180 and 220°C, therefore, the data in Table 3 indicates that the thermal stabilities of these polymers are in the order $IV_b > IV_a > V_b > V_a > IV_c > V_c > IV_d > V_d$. It should be noted that the aromatic based polyesters $IV_{a,b}$ and $V_{a,b}$ are more thermally stable than the aliphatic based polyesters $IV_{c,d}$ and $V_{c,d}$. In particular, the inclusion of the 4-phenylcyclohexanone moiety as a pendant group into the polymer chain increases the thermal stability of these polymers.

4 Conclusions

Linear unsaturated polyesters based on diarylidene-4-phenylcyclohexanone derivatives have been prepared. An interfacial polycondensation synthesis technique at ambient temperature was used. The majority of the polyesters were soluble in aprotic polar solvents and H_2SO_4. Thermogravimetric analyses showed that the aromatic based polyesters were somewhat more thermally stable than their aliphatic counterparts. X-ray diffraction analyses showed that the inclusion of pendant group in the polymer chain causes a decrease in the degree of crystallinity.

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