justify the observed hypsochromic and bathochromic shifts (see Figure 17). We emphasize, however, that the link between hydration level and chromatic switching may involve other structural factors.

In addition to the experimental results reported here, we have observed a broad range of chromatic activity in PXV's and their blends with other polymers. These observations cover essentially the entire spectrum of visible light and are certainly complex functions of molecular environment. Further work is necessary in this area, and we expect to report on it in the future.

Conclusions

Macroion-counterion charge-transfer interactions in solid poly(xylyl viologens) (PXV's) give rise to visible light absorption. The wavelength and intensity of absorption depend not only on conjugation of the cationic backbone and the nature of the counterion but also on the degree of hydration in the solid polyelectrolyte. Water molecules are closely bound to the charged repeating unit of PXV-Br_2 and therefore affect the charge-transfer states of the backbone. This is manifested by a reversible bathochromic shift during thermally induced dehydration. Counterion condensation and the presence of amorphous regions in these semicrystalline polyelectrolytes are important variables in the intensity of light absorption.

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Registry No. PXV-Cl_2 (SRU), 31586-24-0; PXV-Cl_2 (copolymer), 31533-65-0; PXV-Br_2 (SRU), 38815-69-9; PXV-Br_2 (copolymer), 32168-10-8; PXV-I_2 (SRU), 101979-38-8; PXV-I_2 (copolymer), 101979-40-2; PBV-Br_2 (SRU), 68842-39-7; PXPV-Br_2 (copolymer), 101979-41-3; PXPE-Br_2 (SRU), 80485-82-1; PXPE-Br_2 (copolymer), 101979-42-4; DBV-Br_2, 27768-49-6; 4,4'-bipyridine, 553-26-4; α,α'-*p*-dibromoxylene, 623-24-5; (4,4'-bipyridine)·(α,α'-*p*-dibromoxylene)·(sodium acrylate) (copolymer), 101979-43-5.

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Thermotropic Liquid Crystalline Polymers with Quasi-Rigid Chains. 1. Cyclohexyl Moieties

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ABSTRACT: Quasi-rigid polyesters and polyamides with cyclohexyl moieties in their main chains were prepared by solution polymerization. In these polymers the cyclohexyl units retained the configuration they had in the monomers. The 1,4-*trans*-cyclohexyl moieties preserved the linearity of the macromolecules, increased their flexibility, and decreased their packing density. Thus, fusible nematic polymers could be obtained. The incorporation of a mixture of *cis*- and *trans*-cyclohexyl moieties in the polymer chains also led to nematic phases although the cis isomer is nonlinear. However, the mesophase disappeared with increasing cis content.

Introduction

During the search for thermally stable polymers, the nontractable aromatic polyamides [e.g., poly(1,4-phenyleneterephthalamide)] were prepared.[1] Fibers spun from their solutions in concentrated sulfuric acid were found to have high modulus and high strength. This was attributed to the fact that these solutions possess lyotropic mesophases above a certain concentration, which lead to

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oriented extended polymer chains in the fibers.[2,3] Since then, a great effort has been made to synthesize thermotropic liquid crystalline quasi-rigid rodlike polymers in order to avoid the use of corrosive solvents. A large part of this work has been recently reviewed.[4-6] Unfortunately, most of the prepared homo- and copolymers either are nontractable or partially decompose during melting. Many of the fusible copolyesters also reorganize at their high processing temperatures, giving rise to nontractable segments. This hinders an optimal orientation of the polymer chains and leads to inferior mechanical properties.

To lower the melting point (T_m) of rodlike (high axial ratio) polymers below their decomposition temperature, flexible segments, kinks, lateral substituents, or monomers with different dimensions were introduced in the main chains.[7] Quite often these modifications led to the disappearance of the mesophase or to inferior mechanical properties. It is known that the melting temperature of crystalline polymers generally depends on the flexibility of their chains as well as on their packing density.[8] Therefore, it should be expected that replacing some of the phenyl groups in aromatic polymers by cyclohexyl moieties can lower their melting temperatures. In low molar mass liquid crystals, it has been shown that in contrast to the predictions of the Maier–Saupe theory[9] aliphatic rodlike molecules can have quite stable mesophases.[10] In other words, the introduction of 1,4-*trans*-cyclohexyl moieties in the polymer chain would not necessarily lead to the disappearance of the mesophase. Indeed, rodlike polyesters and polyamides containing cyclohexyl units have already been described,[11-17] but little attention has been paid to the isomerization of the cyclohexane ring during polymerization.[11] Many of the data given in the patent literature are also vague and the effect of the cycloaliphatic moiety on T_m is unclear. Therefore, we studied the effect of incorporating cyclohexyl moieties in the main chain of rodlike polyesters and polyamides on their melting temperatures and mesophase stability.

Results and Discussion

One of the problems encountered in the preparation of cyclohexyl polymers is the possible isomerization (cis/trans) of the cycloaliphatic unit, specially under the conditions used in the melt polymerization.[11,18,19] Therefore, we chose the solution polymerization method which can be carried out at low temperatures. The degree of isomerization of 1,4-*trans*-cyclohexanedicarboxylic acid, of its cis–trans mixture (1:1), and of 1,4-*trans*-cyclohexenediol during esterification was determined by GLC in model compounds. It was found that on esterification of 1,4-*trans*-cyclohexanoyl dichloride with ethanol in presence of pyridine at 0–80 °C, 14% of the cis isomer was formed. On esterification of the same acid chloride or of the 1:1 cis–trans mixture under the same conditions with cyclohexanol or phenol, no isomerization occurred. The esterification of 1,4-*trans*-cyclohexanediol with aliphatic or aromatic acid chlorides also led to the pure trans esters. Therefore, it can be expected that the cyclohexyl units in quasi-rigid polymers (no aliphatic primary alcohols) prepared by solution polymerization (Tables III and IV) will retain the configuration they had in the monomers. However, the solution polycondensation is known to lead to low molecular weights in rigid polymers, probably due to their limited solubilities.

In mesomorphic semiflexible polymers, it has been shown that the phase transition temperatures depend on the molecular weight.[20] They increase with increasing degree of polymerization ($\bar{X}$) until they reach maximal values at $\bar{X}$ = 10. For quasi-rigid polymers, the effect of $\bar{X}$ on the phase transition temperatures has not been studied yet. Therefore we prepared poly(1,4-*trans*-cyclohexyl bromoterephthalate) (P29) with different $\bar{X}$ by varying the molar ratio of the monomers. The number-average molecular weights ($\bar{M}_n$) of these polymers were determined by vapor pressure osmometry in 2-chlorophenol at 80 °C. Their $\bar{X}$, intrinsic viscosity [η], and inherent viscosity η_{inh} in the same solvent at 25 °C as well as their melting points are given in Table I. As can be seen, T_m increases by 5 °C per recurring unit in samples a, b, and c ($\bar{X}$ = 6–8) while T_m of P29u ($\bar{X}$ = 14) is 15 °C higher than that of sample a ($\bar{X}$ = 8). Therefore, it can be expected that the relationship between T_m of quasi-rigid polymers and their $\bar{X}$ is quite similar to that of the semiflexible ones. The nematic isotropic transition temperatures of these polymers are higher than their decomposition temperature and, therefore, could not be determined.

Table I
Properties–Molecular Weight Relationship of Poly(1,4-*trans*-cyclohexyl bromoterephthalate)[a]

sample code	[η], mL g^{-1}	η_{inh}, dL g^{-1}	M_n	$\bar{X}$	thermal behavior
P29u	44	0.52	4600	14	C 285–>300 N
P29a	26	0.25	2600	8	C 270–290 N
P29b			2300	7	C 265–285 N
P29c	24	0.23	2000	6	C 260–280 N

[a] $\bar{X}$ is the average number of recurring units. [η] is the intrinsic viscosity. η_{inh} is the inherent viscosity at 25 °C for 0.5% concentration $\bar{M}_n$ is the number-average molecular weight. C indicates crystalline; N, nematic. Temperatures are given in degrees Celsius. The letter u denotes that the polymer was purified by reprecipitation.

Table II
Solution Properties of Some Rodlike Polyesters Prepared by the Solution Polymerization Method[a]

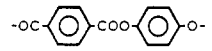
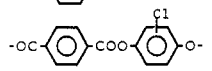
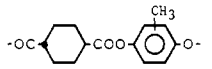
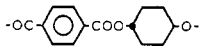
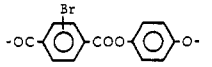
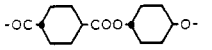
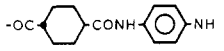
sample code	$\bar{X}$	[η], mL g^{-1}	η_{inh}, dL g^{-1}
P32u	17	56	0.53
P29u	14	44	0.52
P2u	11	20	0.19
P4u	16	22	0.22
P13u	14	17	0.16

[a] For symbols and chemical structure see Tables I and III. [η] at 25 °C, η_{inh} for 0.5% concentration. $\bar{X}$ was calculated from $\bar{M}_n$ determined by vapor pressure osmometry.[21] All measurements were carried out in 2-chlorophenol.

The average number of recurring units in different quasi-rigid polyesters, prepared by condensing equimolar quantities of the monomers in solution, as calculated from their $\bar{M}_n$ are given in Table II. A more detailed account of their solution properties has been given elsewhere.[21] From Table II, it is evident that $\bar{X}$ of these polymers is small and they may better be called oligomers. However, in all cases $\bar{X}$ is higher than 10. Therefore, the thermal data of rodlike polyesters prepared by solution polymerization can be safely compared regardless of $\bar{X}$. The thermal behavior of the investigated quasi-rigid polymers is shown in Tables III and IV.

Poly(1,4-phenyleneterephthalate) P33 is known to be nontractable. Replacement of terephthalic acid in this polymer by 1,4-*trans*-cyclohexanedicarboxylic acid did not help to improve this property, and P30 decomposed before its melting point was reached. The same result was also obtained for P1, where the hydroquinone was replaced by 1,4-*trans*-cyclohexanediol. However, substitution of terephthalic acid in P44 by the hexahydro derivative (P31) led to a thermotropic nematic polymer. The corresponding methyl derivative (P32u) also melted above 290 °C to give a nematic phase. Both nematic phases were identified by

Table III
Influence of the *trans*-Cyclohexyl Moiety on the Thermal Behavior of Rodlike Polyesters and Polyamides[a]

sample code	recurring unit	thermal behavior	remarks
P33		nontractable	decompose above 350 °C
P30		nontractable	decompose above 350 °C
P44		nontractable	decompose above 300 °C
P31		C 260–280 N	miscible with the nematic phase of CBC 33
P32u		C 290–>300 N	miscible with the nematic phase of CBC 33, soluble in ClPh
P1		nontractable	decompose above 300 °C
P43		nontractable	decompose above 350 °C
P29u		C 285–>300 N	miscible with the nematic phase of CBC 33, soluble in TCE and ClPh
P2u		nontractable	decompose above 300 °C, soluble in ClPh
P3		nontractable	decompose above 450 °C
P47		nontractable	decompose above 450 °C
P46		nontractable	decompose above 450 °C

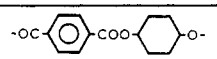
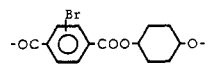
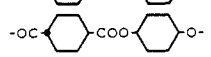
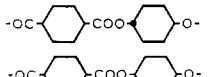
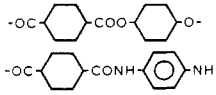
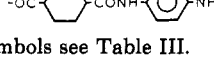
[a] C indicates crystalline; N, nematic; I, isotropic, ClPh, 2-chlorophenol; TCE, *sym*-tetrachloroethane; THF, tetrahydrofuran. Temperatures are given in degrees Celsius and the letter u in the sample code denotes that the polymer was purified by reprecipitation. CBC 33 = 4,4′-bis(4-*trans*-propylcyclohexyl)biphenyl S 220 N 325 I.

their high fluidity and schlieren texture with $s = {}^1/_2$ singularities.[22] Thye were also completely miscible with the nematic phase of 4,4′-bis(4-*trans*-propylcyclohexyl)biphenyl (CBC 33). Replacement of the hydroquinone rest in P43 by a 1,4-*trans*-cyclohexanediol moiety (P29u) also gave a nematic phase above 285 °C. Therefore, it can be said that the 1,4-*trans*-cyclohexyl moiety retains the linearity of the macromolecules and increases its flexibility. Moreover, the packing density of the polymer chains consisting of flat phenyl groups and staggered cyclohexyl units is expected to be less than that of the completely aromatic ones. However, it seems that the cyclohexyl moiety alone is not able to depress the melting point below 300 °C and a lateral substituent is also necessary. The completely cycloaliphatic polymer (P2u) was nontractable, probably due to its harmonious geometry leading to high packing density and due to the absence of lateral substituents. In the polyamides where the hydrogen bonding increases T_m, the same substitution (P46, P47) was not able to depress the melting point enough to give a fusible polymer.

The 1,4-*cis*-cyclohexyl moiety is nonlinear, and the presence of a small amount of the cis isomer in a mesogenic *trans*-cyclohexyl derivative usually leads to the disappearance of the mesophase in low molar mass compounds. However, the effect of incorporating *cis*-cyclohexyl units in a rodlike polymer chain on the mesophase stability is not yet established. Therefore, we studied its effect on the melting point and mesophase stability of polyesters and polyamides although it represents a departure from the rodlike configuration. P4u (Table IV), whose chains contain a mixture of *cis*- and *trans*-cyclohexyl moieties (1:1), melted above 290 °C to give a nematic phase. This is a surprising result and is in contrast with the behavior of the trans isomer P1, which is infusible. Increasing the amount of the cis isomer (P71, P72, P63) lowered the melting point further, but no mesophase could be observed. The same observations were made in P13u and P60u. P57u also melted 85 °C lower than P29u due to the presence of the cis isomer, but the mesophase disappeared. Therefore, it can be said that the *cis*-cyclohexyl unit destabilizes the mesophase more than it depresses the melting temperature. However, nematic phases could be observed in P4u and P13u probably due to the high clearing point of the corresponding trans isomers (high axial ratio and rigidity). The *cis*/*trans*-polyamide P24 was found to be still infusible.

It can be concluded that the linear 1,4-*trans*-cyclohexyl moieties increase the flexibility of the macromolecules and may decrease their packing density. This lowers the

Table IV
Influence of *cis*- and *trans*-Cyclohexyl Moieties on the Mesomorphic Behavior of Quasi-Rigid Polyesters and Polyamides[a]

sample code	recurring unit	cis–trans ratio	thermal behavior	remarks
P4u		1:1	C 290–>300 N	miscible with the nematic phase of CBC 33, soluble in TCE and ClPh
P71u		3:2	C 230–250 I	soluble in TCE and ClPh
P72u		7:3	C 210–230 I	soluble in TCE and ClPh
P63u		6:1	C 180–190 I	soluble in TCE and ClPh
P57u		1:1	C 200–220 I	soluble in THF
P13u		1:1	C 295–>300 N	miscible with the nematic phase of CBC 33, soluble in TCE
P60u		6:1	C 140–160 I	soluble in THF
P65u		1:1	>300	soluble in TCE
P58u		a, 1:1; b, 1:1	C 225–260 I	soluble in THF
P24		1:1	nontractable	decompose above 450 °C

[a] For symbols see Table III.

melting point of quasi-rigid rodlike polymers without destroying their mesophases. *cis*-Cyclohexyl units lower the melting temperature but also destabilize the mesophase so that the mesophase disappears with increasing cis content. However, nematic phases could be still observed at low cis content.

Experimental Section

Characterization of the Polymers: The thermal behavior of the polymers was investigated by differential thermal analysis and polarizing microscopy under N_2 using a PE-DSC2 and a Leitz Orthoplan microscope equipped with a Mettler FP 5/52 heating stage. The transition temperatures were measured under the microscope at a 2 °C/min heating rate, while the thermal analysis was carried out at a rate of 10 °C/min. The upper limit of the heating stage temperature range is 300 °C. The solution viscosities of the polymers were measured with a Schott–Ubbelhode type viscometer at 25 °C (±0.01) for polymer concentrations ranging between 0.5 and 0.15 wt % in 2-chlorophenol. The number-average molecular weights were measured by vapor pressure osmometry in 2-chlorophenol at 80 °C.

General Method of Preparing the Polyesters: A solution of the acid chloride (0.02 M) in 20 mL of dry *sym*-tetrachloroethane (TCE) was added to a cooled solution of the diol (0.02 M) in 15 mL of TCE and 8 mL of dry pyridine under N_2 with stirring. After 30 min, the temperature was left to rise to room temperature and kept there for another 30 min. The reaction mixture was then heated at 70 °C for 2 h, after which it was diluted to 7% concentration and poured on methanol. The so-obtained polymer was washed with methanol and dissolved in TCE or THF when possible. The polyester was reprecipitated in methanol, washed, and dried at 70 °C under reduced pressure.

General Method of Preparing the Polyamides: A solvent mixture consisting of 1 part hexamethylphosphoramide, 2 parts 1-methyl-2-pyrrolidone and 1.5 parts *N,N*-dimethylacetamide was used in this reaction and will be designated PPA. A solution of the acid chloride (0.02 M) in 20 mL of dry PPA was added to a cooled mixture of the diamine (0.02 M), 20 mL of PPA, 8 mL of dry triethylamine and 3 g of lithium chloride under N_2 with stirring. After 30 min at 0–5 °C, the temperature was left to rise to room temerpature and kept there for 1 h. The reaction mixture was diluted with PPA to 7% concentration and then poured on ethanol, and the precipitated polymer was filtered, washed with ethanol, and dried.

1,4-*trans*-Cyclohexanediol: The commercially available product is a 1:1 cis–trans mixture. The trans isomer was separated by recrystallizing the acetyl derivative once from hexane and once from ethanol followed by hydrolysis.[23] The cis-enriched mother liquor was used to prepare the cis-rich polymers. The products were analyzed by GLC on a 2-m 3% Silar 10C column.

1,4-*trans*-Cyclohexanedicarboxylic Acid: The commercial product was crystallized from ethanol until it was 99.5% pure. For the GLC analysis the product was silylated with phenyldimethylchlorosilane prior to injection on a 1-m 3% OV17 column.

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Registry No. P33 (SRU), 26637-45-6; P33 (copolymer), 52871-58-6; P30 (SRU), 102233-56-7; P30 (copolymer), 102233-42-1; P44 (SRU), 9070-65-9; P44 (copolymer), 36424-85-8; P31 (SRU), 58071-35-5; P31 (copolymer), 102233-43-2; P32u (SRU), 66038-74-2; P32u (copolymer), 102233443; P1 (SRU), 102233-57-8; P1 (copolymer), 102233-45-4; P43 (SRU), 95978-14-6; P43 (copolymer), 102233-46-5; P29u (SRU), 102282-99-5; P29u (copolymer), 102233-47-6; P2u (SRU), 88392-10-3; P2u (copolymer), 88392-15-8; P3 (SRU), 53467-77-9; P3 (copolymer), 53504-12-4; P47 (SRU), 102283-01-2; P47 (copolymer), 102233-48-7; P46 (SRU), 102283-02-3; P46 (copolymer), 102233-49-8; P44 (terpolymer), 102233-50-1; P57u (SRU), 102283-00-1; P57u (terpolymer), 102233-51-2; P13u (terpolymer), 102233-52-3; P65u (terpolymer), 102233-53-4; P58u (quaterpolymer), 102233-54-5; P24 (terpolymer), 102233-55-6; P4u (SRU), 102233-58-9.

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