1270 and 1100 (C–O–C). Compound **3** was obtained as white needles from pentane, mp 27 °C. ^{1}H NMR δ 6.2 and 5.8 (H_a, 4 H), 4.8 (H_f, 2 H), 4.3 (H_c, 4 H), 3.8 (H_e, 6 H). IR (cm^{-1}) 1730 (carbonyl), 1620 (alkene), 1125 and 975 (C–O–C). Compound **4** was obtained as a clear oil. ^{1}H NMR δ 6.2 and 5.8 (H_a, 4 H), 4.8 (H_g, 4 H), 4.2 (H_c, 4 H), 3.7 (H_e, 6 H). IR (cm^{-1}) 1750 (carbonyl), 1610 (alkene), 1125 and 975 (C–O–C). All compounds gave satisfactory elemental analyses.

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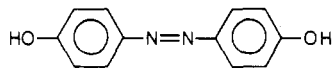
New Polyesters and Polyformals Containing Multiple *p*-Aryleneazo Groups: Liquid-Crystal Polyazoaryl Sebacates

Introduction. Liquid-crystal (lc) polymers have been of great interest in recent years (for recent reviews, see ref 1–4). Lc polymers come in several types: (1) the entire chain is mesogenic; (2) mesogenic groups are in the side chain; (3) mesogenic groups are in main chain. Our study will be confined to polymers in class 3. Polymers based on stilbene derivatives, tolanes, and aromatic polyimines have been reported to display lc properties. However, although the *p*-phenyleneazo link is known to favor lc behavior in small molecules, it has only recently been briefly reported to cause lc polymer formation.[5]

Polyesters have been the most popular type of thermotropic lc polymer. Accordingly, in this work we have synthesized a number of aromatic and aliphatic polyesters from bisphenols containing one to three *p*-phenyleneazo groups. The polymers have been examined for their lc properties. Aromatic formals have come into attention recently,[6,7] and we also synthesized them from a number of azobis(phenols).

Results. Monomers: Bis(phenols). The bis(phenol) monomers in this study are described in Table I. In general, they were obtained as indicated by the literature or were synthesized by standard procedures involving aryldiazonium ion couplings.[8,9] Although most of these compounds were reported in the literature (see references included in Table I), none (except 1) offered detailed procedures, elemental analyses, or melting points. Representative synthesis procedures are given in the Experimental Section.



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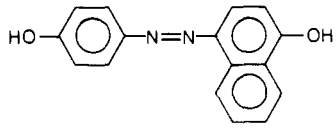

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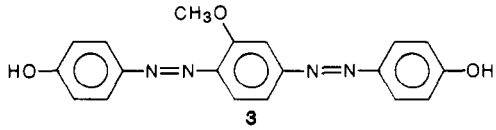

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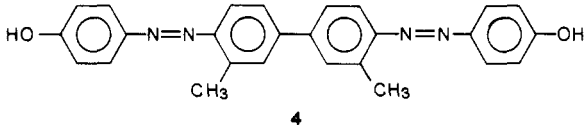

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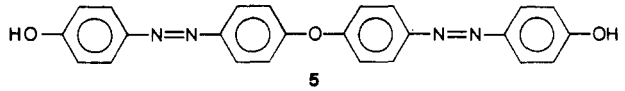

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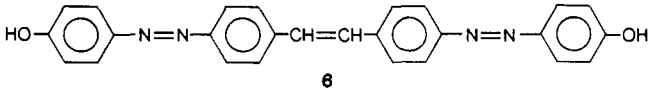

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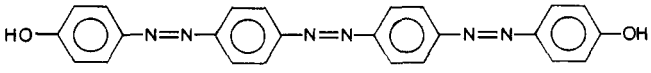

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Diacids. An important factor in these studies was the tractibility of the polymers. We wanted to avoid intractable, infusible "brick dust". Inasmuch as the *p*-phenyleneazo group tends to form intractable polymers, we used relatively "soft" diacyl halides to offset the "hard" bis(phenol). Because the "soft" diacid components possessed meta aromatic or aliphatic structures, the resulting combinations represent a trade-off between lc character and tractability. Isophthaloyl chloride and 5-*tert*-butylisophthaloyl chloride were the aromatic acid chlorides employed. As an aliphatic diacid, sebacic acid was used.

Polymerizations. All of the polyesters were synthesized by interfacial polycondensation. The results are given in Table II.

Polyformals were synthesized by use of excess dichloromethane in *N*-methylpyrrolidone solution as described in the literature.[7]

Properties of Polymers. The properties of the polymers we synthesized are collected in Table II. These include data on polymer melting behavior, inherent viscosities, film-forming ability, color, and lc behavior.

Polymer solubility was increased by the 5-*tert*-butyl group in isophthalic acid and the methoxy group in **3**. Derivatives of the bis(phenols) **2** and **5** had the greatest solubility. This factor helped us to achieve a higher molecular weight by keeping the polymer in solution longer. Films were cast from the polymer solution by evaporating the solvent. For those chloroform-soluble polymers, GPC chromatograms were obtained. They showed broad molecular weight distribution with the highest concentration at MW 14 000 for **2**-5-*t*-BuI, 10 000 for **3**-Seb, and 35 000 for **5**-5-*t*-BuI.

LC Behavior. The occurrence of lc behavior depended strongly on the bis(phenol) monomer and on the diacid. Both small molecules (Tables I and III) and polysebacates (Table II) based on **4** and **7** uniformly showed lc characteristic melting point behavior. Polysebacates based on **2**, **3**, and **5** also showed lc properties. On the other hand, monomer or derivatives of **5** showed no lc behavior. All the liquid-crystalline compounds and polymers, as listed in Table I–III, showed nematic textures. Some of the optical textures of these small molecules and polymers in the liquid-crystal state, as observed with a hot-stage polarizing microscope, are shown in Figures 1 and 2.

Discussion. We have synthesized new thermotropic liquid-crystal polyesters based on various azobis(phenols). Polyformals were also synthesized from these monomers. The polymers were fusible and soluble in organic solvents and hence were processable. They represent a novel class of polymers.

Thermotropic lc behavior was found for the polysebacates of **2**, **3**, **4**, **5**, and **7**. On the other hand, no lc properties were displayed by any of the polyisophthalates or poly(5-*tert*-butylisophthalates). Weak lc behavior was seen for the polyformal of **4**.

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Table I
Monomer Synthesis and Properties

monomer	synth react	recryst solvent	yield color	yield %	mp,[a] °C	elem anal. calcd	elem anal. found	ref
1	diazotization coupling	toluene	black	30	215, lit. 218			10
2	diazotization coupling	benzene or 50% ethanol	black green	86	dec 180	C, 72.73; H, 4.55; N, 10.61	C, 72.24; H, 4.25; N, 8.32	11
3	repeated diazotization coupling	benzene	red-brown	30	tm 99, dec 136 (lc)	C, 65.52; H, 4.60; N, 16.09	C, 63.50; H, 4.30; N, 15.54	
4	tetraazotization	toluene	brown	83	tm 227, ti 250 (lc)	C, 73.93; H, 5.21; N, 13.27	C, 73.18; H, 5.20, N, 12.67	12
5	tetraazotization coupling	acetic acid	yellow-brown	85	dec 260	C, 70.24; H, 4.39; N, 13.66	C, 69.92; H, 4.34; N, 13.49	13
6	tetraazotization coupling	acetic acid	dark green	80	>300	C, 74.29; H, 4.76; N, 13.33	C, 73.13; H, 4.36; N, 12.77	14
7	tetraazotization coupling	95% ethanol	dark green	42	tm 276, dec 286 (lc)	C, 68.25; H, 4.27; N, 19.91	C, 67.52; H, 4.15; N, 19.33	15

[a] tm, transition from the solid to the mesophase; ti, transition from the mesophase to the isotropic liquid phase.

These results agree with the generally accepted idea that extended rodlike character is required for lc behavior. Only the sebacate group, with its ability to adopt the trans, planar, extended zig-zag form, meets this criterion. The phthalate residue, as well as the methylene formal units, lead to kinks in the chain. These disrupt the propensity for lc character shown by the monomeric bis(phenols) and their simple derivatives.

Experimental Section. Methods. ^1H and ^{13}C NMR spectra were taken on a 60-MHz Varian EM 360L or 250-MHz Bruker WM 250 spectrometer. Infrared spectra were recorded on a Perkin-Elmer 983 spectrometer. Molecular weights of the polymers were estimated by gel permeation chromatography (columns, Du Pont Zorbax PSM-300S, Du Pont Zorbax PSM-60S, IBM 10-μm pore; detector, Spectra-Physics 8200 UV (254 nm) detector; eluent, chloroform). Inherent viscosity was determined by using an Ostwald-type viscometer in a constant-temperature bath. Elemental analyses were performed by Mic Anal, Tucson, AZ.

Diazotization and Coupling. To a stirred solution of 4.36 g (0.04 mol) of *p*-aminophenol in water was added 12 mL (0.14 mol) of concentrated HCl. The mixture was then cooled in an ice bath and a solution of 2.9 g (0.0404 mol) of sodium nitrite was added. The mixture was tested for the presence of free nitrous acid with potassium iodide–starch paper after 15 min. To a stirred solution of 7.2 g (0.05 mol) of 1-naphthol and 4.8 g (0.12 mol) of sodium hydroxide in ice–water was added the above mixture in a period of 30 min. The reaction mixture was then stirred for 1 h. The resulting greenish brown suspension was acidified with dilute HCl and cooled. The green precipitate was collected on a funnel, dried, and recrystallized from benzene to give 9 g (86%) of 4-[(4-hydroxyphenyl)azo]-1-naphthol (**2**), dec 180 °C.

The compounds synthesized in this work were routinely characterized by IR and NMR in addition to the elemental analysis shown in the tables. The spectral data for **2** and **3** are as follows: **2**, IR (KBr) 3440 cm^{-1}, 1588, 1509, 1472, 1238, 827, 760; ^1H NMR (CD_3COCD_3) δ 7–8 (m); ^{13}C NMR (CD_3SOCD_3) δ 160.0 (C), 156.7 (C), 147.9 (C), 146 (C), 139.5 (C), 132.3 (C), 127.4 (CH), 125.3 (CH), 124.4 (CH), 122.7 (CH), 115.9 (CH), 113.1 (CH), 108.4 (CH); **3**, IR (KBr), 3269 cm^{-1}, 1588, 1504, 1463, 1244, 839; ^1H NMR (CD_3COCD_3) δ 4.05 (s, 3 H), 7–8 (m, 11 H); ^{13}C NMR (CD_3COCD_3) δ 126.0 (CH), 117.8 (CH), 117.0 (CH), 116.7 (CH), 106.5 (CH), 56.6 (CH_3).

Tetraazotization and Coupling. To a stirred solution of 8.48 g (0.04 mol) of tolidine in water was added 16.8 mL (0.2 mol) of concentrated HCl. The mixture was cooled to room temperature and a solution of 5.84 g (0.0808 mol) of sodium nitrite was added. The mixture was tested for the presence of free nitrous acid with potassium iodide–starch paper after 30 min. To a stirred solution of 9.36 g (0.1 mol) of phenol and 5.6 g (0.14 mol) of sodium hydroxide in ice–water was added the above mixture in a period of 30 min. The reaction mixture was then stirred for 1 h. The resulting yellowish brown suspension was acidified with dilute HCl. The yellow-brown precipitate was collected on a funnel, dried, and recrystallized from toluene to afford 14.06 g (83%) of **4**.

Diacetate. To a solution of 0.132 g (0.5 mmol) of 4-[(4-hydroxyphenyl)azo]-1-naphthol (**2**) in pyridine was added an excess of acetyl chloride dropwise. The reaction mixture was then stirred at room temperature for 30 min. The resulting yellow suspension was poured into water. The yellow precipitate was isolated, washed repeatedly with hot 95% ethanol to give 0.17 g (100%) of **2**-OAc.

Diisobutyrate. To a stirred solution of 0.211 g (0.5 mmol) of bis(phenol-4-azo)-4,4′-(3,3′-dimethylbiphenyl) (**4**) in pyridine was added an excess of isobutyryl chloride. The reaction mixture was then refluxed for 10 h. The resulting mixture was poured into water and extracted with chloroform, the organic layer was separated and dried, and solvent was evaporated and crystallized from 95% ethanol to give 0.09 g (32%) of 4-O-*i*-Bu.

Dimethyl Ether. To a stirred solution of 0.406 g (1 mmol) of 4,4′-oxybis(*p*-phenyleneazo)bis(phenol) (**5**) 0.4 g (10 mmol) of sodium hydroxide, and a catalytic amount of tetrabutylammonium hydrogen sulfate in water was added an excess of dimethyl sulfate. The reaction mixture was refluxed for 2 h. The resulting precipitate was filtered, washed repeatedly with hot ethanol, and dried to give 0.40 g (91%) of 5-OMe, mp 228 °C.

Polyester. A solution of 0.13 g (0.5 mmol) of 5-*tert*-butylisophthaloyl chloride in 20 mL of CH_2Cl_2 was introduced into a blender. To this was added a solution of 0.205 g (0.5 mmol) of 4,4′-oxybis(*p*-phenyleneazo)bis(phenol) (**5**) and 0.041 g (1.01 mmol) of sodium hydroxide in 25 mL of distilled water. The mixture was stirred vigorously for 15 min. The resulting mixture was then poured into 400 mL of methanol with stirring. The yellow precipitate was filtered with suction, washed repeatedly with methanol, and dried in a vacuum to give 0.25 g (84%) of 5-5-*t*-BuI.

Table II
Polymer Synthesis and Properties

bis-(phenol)	di-chloride[a]	react conditns	yield, %	color	appearance, film-forming ability; solubility	η(inh), 30 °C, 0.5 g/dL (solvent)	mp, °C	elem anal. calcd	elem anal. found
1	I	interfacial (CH_2Cl_2, NaOH, H_2O)	57	yellow	powder; H_2SO_4		>300		
	5-*t*-BuI	interfacial	75	light yellow	film; H_2SO_4		>300		
2	I	interfacial	61	red-brown	powder; TFA, H_2SO_4	0.05 (H_2SO_4)	>300		
	5-*t*-BuI	interfacial	80	yellow	film; $CHCl_3$ etc.	0.17 ($CHCl_3$)	dec 323	C, 74.67; H, 4.89; N, 6.22	C, 73.47; H, 4.78; N, 5.22
	Seb	interfacial	97	yellow	film; $CHCl_3$ etc.	0.22 ($CHCl_3$)	tm 98 dec 165 (lc)	C, 72.56; H, 6.05; N, 6.51	C, 69.45; H, 6.20; N, 4.89
	M	NMP; NaOH, Bu_4NI	77	red	film; DMAC, TFA, NMP	0.52 (H_2SO_4)	soft at 200	C, 73.91; H, 4.35; N, 10.14	C, 72.01; H, 4.59; N, 9.44
3	Seb	interfacial	97	orange	film; $CHCl_3$ etc.	0.14 ($CHCl_3$)	tm 108, dec 240 (lc)	C, 67.70; H, 5.84; N, 10.89	C, 65.11; H, 5.62; N, 8.99
4	5-*t*-BuI	interfacial	82	yellow	film; TFA H_2SO_4	0.19 (H_2SO_4)	dec 333	C, 75.00; H, 5.26; N, 9.21	C, 73.91; H, 5.74; N, 9.25
	Seb	interfacial	60	red-yellow	film; TFA, H_2SO_4	0.12 (H_2SO_4)	tm 160, dec 250 (lc)	C, 73.47; H, 6.12; N, 9.52	C, 71.68; H, 6.23; N, 8.37
	M	NMP; NaOH, Bu_4NI	92	red-orange	film; H_2SO_4	0.21 (H_2SO_4)	tm 239,[b] dec 260 (lc)	C, 74.65; H, 5.07; N, 12.90	C, 73.68; H, 4.95; N, 12.08
5	I	interfacial	80	brown	powder; TFA H_2SO_4	0.07 (H_2SO_4)	>300		
	5-*t*-BuI	interfacial	84	yellow	film; $CHCl_3$ etc.	0.56 ($CHCl_3$)	dec 398	C, 72.48; H, 4.70; N, 9.40	C, 70.05; H, 4.34; N, 8.29
	Seb	interfacial	70	yellow	film; TFA H_2SO_4		tm 245, dec 278 (lc)	C, 70.83; H, 5.56; N, 9.72	C, 69.80; H, 5.48; N, 9.96
	M	NMP; NaOH, Bu_4NI	97	yellow	powder; TFA H_2SO_4	0.16 (H_2SO_4)	dec 317	C, 71.09; H, 4.27; N, 13.27	C, 70.45; H, 4.15; N, 12.98
6	I	interfacial	58	brown	powder; H_2SO_4		>300		
	5-*t*-BuI	interfacial	59	brown	powder; TFA H_2SO_4	0.10 (H_2SO_4)	>300		
7	5-*t*-BuI	interfacial	68	dark brown	powder; H_2SO_4		>300	C, 71.05; H, 4.61; N, 13.82	not satisfactory
	Seb	interfacial	68	brown	powder; TFA H_2SO_4	0.09 (H_2SO_4)	tm 244, dec 259 (lc)	C, 69.39; H, 5.56; N, 14.29	C, 66.92; H, 4.97; N, 15.54

[a] I, isophthaloyl; Seb, sebacoyl; 5-*t*-BuI, 5-*tert*-butylisophthaloyl; M, methylene chloride. [b] On pressing at this region, the sample looks nematic but it does not flow.

Table III
Model Compound Synthesis and Properties

model compd	color	yield, %	mp, °C	elem anal. calcd	elem anal. found
2-OAc	light yellow	100	133[a]	C, 68.97; H, 4.60; N, 8.05	C, 68.76; H, 4.66; N, 7.84
4-OAc	orange	93	tm 194, dec 295 (lc)	C, 71.15; H, 5.14; N, 11.07	C, 71.20; H, 4.87; N, 10.97
5-OAc	light yellow	88	246	C, 68.02; H, 4.45; N, 11.34	C, 68.07; H, 4.26; N, 11.40
4-O-*i*-Bu[b]	orange	32	tm 165, dec 281 (lc)	C, 72.60; H, 6.05; N, 9.96	C, 72.17; H, 5.67; N, 9.68
5-O-*i*-Bu	yellow	44	237	C, 69.82; H, 5.45; N, 10.18	C, 68.12; H, 5.30; N, 10.51
4-OMe	orange	73	tm 142, dec 298 (lc)	C, 74.67; H, 5.78; N, 12.44	C, 74.35; H, 5.62; N, 12.50
5-OMe	light yellow	91	228	C, 71.23; H, 5.02; N, 12.79	C, 70.46; H, 4.84; N, 12.46

[a] First heating on DSC showed two sharp peaks at 128 and 138 °C, but no mesophase was observed with a polarizing microscope; on second heating, only one peak at 133 °C was recorded. [b] O-*i*-Bu, isobutyrate.

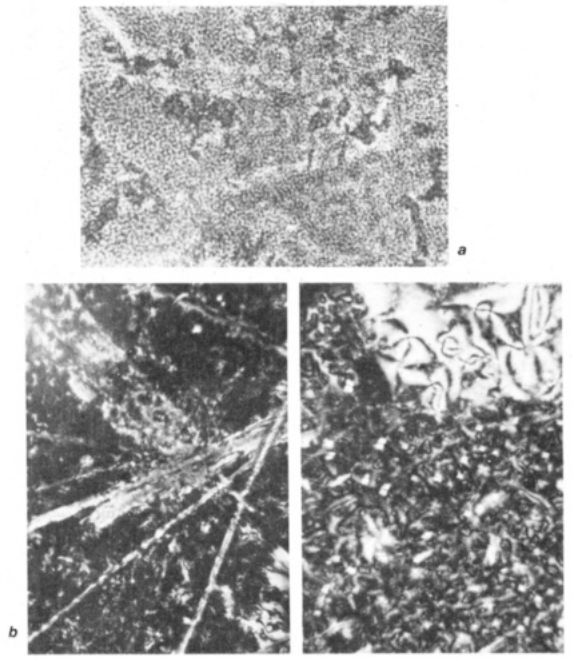

Figure 1. Photomicrograph of the small molecules: (a) **3**, taken at 130 °C; (b) 4-OAc, taken at 196 °C; (c) 4-OAc, taken at 280 °C (magnification of 128×).

Polyformal. Into a 50-mL round-bottom flask equipped with a magnetic stirrer and a reflux condenser were added 0.422 g (1 mmol) of bis(phenol-4-azo)-4,4′-(3,3′-dimethylbiphenyl) (**4**), 3 mL of CH_2Cl_2, 10 mL of *N*-methylpyrrolidone, 0.09 g (2.02 mmol) of NaOH, and 0.738 g (2 mmol) of tetrabutylammonium iodide. The mixture was refluxed under nitrogen for 24 h. After cooling, the resulting mixture was poured into 300 mL of methanol with stirring and neutralized with dilute acetic acid. The red-orange precipitate was then filtered, washed repeatedly with methanol, and dried under vacuum to give 0.4 g (92%) of 4-M.

Characterization of the Liquid-Crystalline Compounds and Polymers. All the thermal transition temperatures were recorded on a Perkin-Elmer DSC 4 or DSC 2 at a heating rate of 20 °C/min under nitrogen atmosphere. The optical textures were observed on a hot-stage (Mettler FP-2 or FP-82) polarizing microscope (Leitz, Ortholux) at a magnification of 320×.

Acknowledgment. We are deeply indebted to the Office of Naval Research for partial support of this work.

Registry No. (1)(I) (copolymer), 25215-81-0; (1)(I) (SRU), 24938-83-8; (1)(5-*t*-BuI) (copolymer), 108796-15-2; (1)(5-*t*-BuI) (SRU), 108796-32-3; **2**, 51828-71-8; 2-OAc, 108796-08-3; (2)(I) (copolymer), 108796-16-3; (2)(5-*t*-BuI) (copolymer), 108796-17-4; (2)(M) (copolymer), 108796-19-6; (2)(Seb) (copolymer), 108796-18-5; **3**, 108796-06-1; (3)(Seb) (copolymer), 108796-20-9; (3)(Seb) (SRU), 108834-40-8; **4**, 108796-07-2; 4-OMe, 108796-14-1; 4-OAc, 108796-11-8; 4-O-*i*-Bu, 108796-09-4; (4)(5-*t*-BuI) (copolymer), 108796-21-0; (4)(5-*t*-BuI) (SRU), 108796-33-4; (4)(Seb) (copolymer), 108796-22-1; (4)(Seb) (SRU), 108796-34-5; (4)(M) (copolymer), 108796-23-2; (4)(M) (SRU), 108796-35-6; **5**, 30546-14-6; 5-OMe, 108796-10-7; 5-OAc, 108796-12-9; 5-O-*i*-Bu, 108796-13-0; (5)(5-*t*-BuI) (copolymer), 108796-25-4; (5)(5-*t*-BuI) (SRU), 108796-37-8; (5)(I) (copolymer), 108796-24-3; (5)(I) (SRU), 108796-36-7; (5)(Seb) (copolymer), 108796-26-5; (5)(Seb) (SRU), 108796-38-9; (5)(M) (copolymer), 108796-27-6; (5)(M) (SRU), 108796-39-0; (6)(5-*t*-BuI) (copolymer), 108796-29-8; (6)(5-*t*-BuI) (SRU), 108796-41-4; (6)(I) (copolymer), 108796-28-7; (6)(I) (SRU), 108796-40-3; (7)(5-*t*-BuI) (copolymer), 108796-30-1; (7)(5-*t*-BuI) (SRU), 108796-42-5; (7)(Se) (copolymer), 108796-31-2; (7)(Se) (SRU), 108796-43-6; 4-$H_2NC_6H_4OH$, 123-30-8; C_6H_5OH, 108-95-2; 1-naphthol, 90-15-3; tolidine, 29158-17-6.

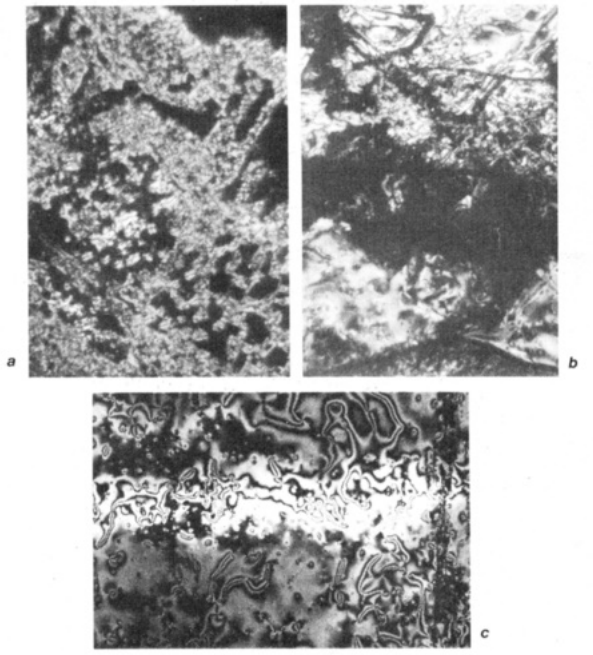

Figure 2. Photomicrograph of the polymers: (a) 4-Seb, taken at 180 °C; (b) 4-Seb, taken at 200 °C; (c) 7-Seb, taken at 250 °C (magnification of 128×).

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