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Syntheses and Investigation of Some New Polyarylates and Copolyarylates. Part I

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ABSTRACT

By the interfacial condensation of acid chlorides I, II, and III, respectively, with bifunctional phenols (IV-X), soluble or moldable thermostable polyarylates were obtained. Similarly, copolyarylates were prepared from a mixture of the acid chlorides I + II or I + III. With bisphenol-A (VIII), soluble polymers are usually obtained. Methylene dichloride and/or carbon tetrachloride-water systems were the best media for interfacial condensations, and the polymers formed showed the highest reduced viscosity values.

Polyester films are useful in many industrial applications because of their broad range of mechanical, optical, and electrical properties. The aromatic polyesters—polyarylates—dominate the field of industrial polyester films [1].

Polyarylates prepared from dihydric phenols and purely aromatic dicarboxylic acid chlorides are highly heat resistant materials [2], but they are insoluble and nonmoldable [3]. However, by the introduction of ether linkages in the aromatic dicarboxylic acid moiety, soluble and/or moldable polyamides were obtained [4].

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In the present work, preliminary investigation showed that melt condensation of amino and/or dihydric phenols with terephthaloyl chloride (I), 4,4'-bis-(chloroformylmethoxy) diphenyl (II), and bis-(4-chloroformylmethoxyphenyl) sulfone (III) is unsuccessful. Consequently, interfacial polycondensation was attempted and a series of homopolyarylates and polyamidoarylates were obtained by the interaction of equimolar amounts of acid chlorides [5, 6] I, II, and III [7], respectively, with quinol (IV), resorsinol (V), 3-aminophenol (VI), 4-aminophenol (VII), 2,2-bis-(4-hydroxyphenyl) propane (VIII) [8], bis-(4-hydroxyphenyl) sulfone [9], and 4,4'-dihydroxydiphenyl [8]. The condensation was effected in a binary phase system: watersolvent immiscible system. The solvents used were benzene, methylene dichloride, chloroform, and carbon tetrachloride [10-13] in the presence of sodium hydroxide [5].

Table 1 lists the data relevant to reduced viscosity values and percentage yields of the polymers prepared from acid chloride III with different phenols (IV-X). Close study of the table shows that bis-(4chloroformylmethoxyphenyl) sulfone-polyarylates and polyamidoarylates have high viscosities in different organic solvents, evidently due to good dissolution of these polymers [11, 12].

It has been claimed that peaks in polymer viscosities arise from variations of reactant concentrations [14, 15], and highest viscosities of the polymers synthesized were noted at acid chloride concentrations ranging from 0.04 to 0.02 mol/L. The same result was confirmed in the present investigation, and using higher acid chloride concentrations led, in most cases, to lower viscosities. This result is not unexpected since it can be explained in terms of partition coefficients changes at different concentrations [16]. Further, it finds support by previous work [5, 13, 14] on the synthesis of some aromatic polyamides [17]. Moreover, the low viscosity values and yields obtained at high reactants concentrations (Table 1) may be ascribed to the formation of an oligomer phase which captures a quantity of the solvent, thus disturbing the mass intensity transfer process. This in turn distrubs, restricts, and slows down the diffusion of the rigid aromatic acid chloride molecules through the primarily formed compact polymer film. With high acid chloride concentrations the amount of the captured solvent is obviously so high that it gives a gelatinous mass which effectively stops or greatly lowers the stirring rate. In effect, this criterion was noticed in the case of acid chloride III with phenols VI and VIII, respectively (Table 1).

The role of solvents in interfacial polymer formation has been extensively studied, and quasi-solvents proved to be effective through solubility differences [11, 12]. In the present work, methylene dichloride and carbon tetrachloride can be considered as polymers near solvents since the highest polymers yields and viscosities were noted with them (Table 1). These solvents probably enhance swelling of the polymer film at the boundary surface and consequently the mobility of the acid chloride molecules through their reaction with the phenoxide ion in the aqueous phase [11, 16]. If the polymer film is otherwise

TABLE 1.	Effect of §	solvent	and Rea	ctants	Concer	itratio	n on Vis	scositi	es (η_{re})	d and	Yields	(%) of	Polyar	ylates	and
Polyamidoaı	rylates Ot	otained	from Ac	sid Chl	oride I	II and	Differen	nt Bisp	henols	50					
			()1	E	(2	(1 3-Ar	(L) -onin	(V An	П) ino-	(V 2,2-I hydr nhe	III) Bis(4- 'oxy- nvl)	(L Bis hydr	K) (4- oxy-	() 4,4'. hvdr	۲) -Di- 0XV-
	Reactan	Ъ С	linol	Resol	cinol	phe	lon	phe	lou	lord	pane	sulf	one	diph	enyl
Solvent	tration	η_{red}	Yield	η_{red}	Yield	η_{red}	Yield	η_{red}	Yield	$\eta_{\mathbf{red}}$	Yield	η_{red}	Yield	η_{red}	Yield
Benzene	0.1									0.14	79.2				
	0.04	0.14	36.7	0.07	41.9	0.08	54.2	0.11	40.7	0.16	79.3	0.08	65.7	0.14	56,9
	0.02	0.14	16.4	0.08	27.2	0.17	61.6	0.18	52.4	0.22	65,6	0.06	73.5	0.25	45.9
	0.01	0.15	13.9	0.09	59.8	0.27	83.3	0.16	52.7	0.29	61.4	0.05	65.2	0.18	50.3
Methylene	0.1					0*0	50.7								
chloride	0.04	0.15	39.5	0.05	75.9	0.14	66.4	0,09	60.3	0,09	6° 96	0.09	91.1	0.23	84.4
	0.02	0.13	55.7	0,06	69,3	0.22	81,1	0.11	72.4	0.18	95.0	0.10	74.2	0.26	88.9
	0.01	0.11	23.3	0.13	67.0	0.28	84.3	0.14	62.4	0.14	88.1	0,09	68,1	0.24	79.9
Chloroform	0.04	0.15	18.6	0.06	73.1	0.11	73.8	0.13	56.3	0.15	67.8	0.16	78.9	0.19	81.1
	0.02	0.20	26.8	0.12	62.6	0.20	82.1	0.17	73.6	0.16	73,2	0.27	79.1	0.21	59.9
	0.01	0.15	18.3	0.08	55,6	0.32	6° 96	0.24	80.7	0.17	84.9	0.27	67.8	0.18	72.6
Carbon	0.04	0.04	39,3	0.16	45.1	0.07	54.7	0.07	36.6	0.17	93.4	0.15	36.8	0.16	36.5
tetra- chloride	0.02	0.06	26.8	0.22	44.2	0.08	40.3	0.10	32.0	0.18	72.5	0.23	53,6	0.16	44.2
	0,01	0.04	18.3	0.20	48.5	0.10	68.8	0.12	69.3	0.19	76.4	0.16	47.6	0.17	69.4
$a_{\eta_{red}}$ (re	sduced vis	cosity)	measur	ed in (dimethy	lform	amide.								

TABLE 2. Effect of Solvents on Viscosities and Yields of Polymers Synthesized from Terephthaloyl Chloride (I) and 2,2-Bis-(4-hydroxyphenyl) Propane (VIII) (1:1)

Solvent	Carbon tetrachloride	Methylene chloride	Chloroform	n-Hexane	Benzene	Nitro- benzene
η_{red}^{a}	0.85	0.74	0.57	0.58	0,58	0.46
% yield	93.5	96.0	97.7	88.3	95.4	96.1

 a_{r}^{a} red measured in phenol-tetrachloroethane mixture (6:4).

TABLE 3. Effect of Solvents on Viscosity and Yields of Copolymers Synthesized from Terephthaloyl Chloride (I) + 4, 4^t-Bis-(chloroformylmethoxy) Diphenyl (II) with 2, 2-Bis-(4-hydroxyphenyl) Propane (VIII) (1:1)^a

% cor	nposi-				Sol	vents			
chlc chlc mixt	ride ures	Ca	rrbon chloride	Chlo	roform	Met	hylene loride	Ben	zene
I	п	η_{red}	Yield %	η_{red}	Yield $\%$	η_{red}	Yield %	η_{red}	Yield %
90	10	0.65	94.7	0.28	80.5	0,33	83.5	0.31	92.1
80	20	0.54	88.9	0.24	76.4	0.27	78.3	0.25	88.9
70	30	0.36	83.5	0.19	60.4	0.22	71.8	0.20	82.7
60	40	0.28	82.7	0.16	55.4	0.17	68.5	0.12	80.1
a	red mea	asured in	phenol-tetrac	hloroethan	e mixture (6:4	()			

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ride	[)a
Chlo	[] []
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lers	Bis-
olyn	2,2-
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d Yie	fone
y an	l) Sul
cosit	henyl
ı Vis	oxypl
ent or	meth
Solve	rmylı
t of	rofoi
Effec	-chlo
4	s-(4-
BLE	+ Bi
TA	Ξ

% cor sitio	n po- n of					Sol	vents				
ride 1 tur	nix-	Ca. tetrac	rbon hloride	Chlor	oform	Meth chlc	ylene Jride	Ben	zene)H-u	exane
I	日	η_{red}	Yield $\%$	η_{red}	Yield %	η_{red}	Yield %	η_{red}	Yield%	η_{red}	Yield %
90	10	0.60	82.2	0.33	93.2	0.54	91.3	0.35	85,6	0.49	75.2
80	20	0.52	74.1	0.26	91.8	0.46	89,6	0.26	80.2	0.42	66.9
20	30	0.46	72.2	0.21	75.4	0.34	84.3	0.21	77.2	0.32	60.4
60	40	0.38	70.8	0.17	74.5	0.24	72.5	0.17	68.9	0.19	53.8
a	red mea	sured in	phenol-tet:	rachloroe	thane mix	ture (6:4).				

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TABLE 5. Effect of Acid Chloride Concentration on Viscosities and
Yields of Copolymers Synthesized from Terephthaloyl Chloride (L
80%) + Bis-(4-chlorophenylmethoxyphenyl) Sulfone (III, 20%) with 2,2-
Bis-(4-hydroxyphenyl) Propane (VIII; 0.17 mol) in Carbon Tetra-
chloride $(7.5 \text{ mL})^{a}$

. • •

		Acid chlori	ides concent	ration (mol/	Ľ)
	0.17	0.33	0.50	0.66	0.99
$\eta_{\mathbf{red}}$	0.74	0.30	0.22	0.14	0.13
Yield $\%$	42.50	86.6	84.3	82.0	77.2

 ${}^{a}\eta_{red}$ measured in phenol-tetrachloroethane mixture (6:4).

solvent insoluble, obstruction of acid chloride migration would have occurred, thus leading to poor quality polymers [11]. Still, there is no evidence that the interface has any special orienting or aligning effect on the reactants. In the light of the above-mentioned points, one can explain the formation of insoluble polymeric substances by the interaction of acid chloride II with bisphenols IV-X.

In an attempt to improve the properties of the homopolyarylates of the acid chloride II, copolycondensation of a mixture of acid chlorides II + I and II + III with the different phenolic compounds (IV-X)was experienced while being aware of the copolycondensation necessary stipulations [11, 13]. With the exception of 2,2-bis(4-hydroxyphenyl) propane (VIII), all the other phenols yielded insoluble copolyarylates with I + II or I + III acid chloride mixtures. Data relevant to the copolycondensation of VIII with the last-mentioned acid chloride mixtures are summarized in Tables 3 and 4. As expected, the copolyarylates synthesized have amalgamated properties of the two possibly formed, parent homopolyarylates [18]. This becomes quite evident from a study of the temperature dependencies of the polymer mechanical properties upon their transition points. The softening points are known to differentiate a number of physical states which differ substantially from one another in mechanical properties 3. 18-20]. Polymer softening points and polymer thermal stabilities, as manifested by their intensive decomposition temperatures, are given in Tables 6a and 6b and shown by Figs. 1-4. From these tables and figures it is evident that homopolyarylates have intensive decomposition temperatures higher than those for the copolyarylates derived from the same reactants and at the optimum reaction conditions. Moreover, it is quite clear that the replacement of the strongly polar bis-(4-chloroformylmethoxyphenyl) sulfone acidic group for the less polar 4,4'-bis-(chloroformylmethoxy) diphenyl group led to reduction of the thermal stability of the copolymers prepared. Thus it can be

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TABLE 6a. Transition phenols (IV-X) with Ter chloroformylmethoxyph	and Inter rephthalo ienyl) Suli	sive Deco yl Chloride fone (III)	mposition Temper e (I), 4,4'-Bis-(chl	atures of Pol oroformylme	yarylates thoxy) Di _l	Synthesize phenyl (II),	ed from E and Bis-	iis- (4-
			I		Π		Η	
Phenol	mp (°C)	Soften- ing point (°C) ^a	Intensive decompo- sition tem- perature (°C)	Activation energy (kcal/ mol)	mp (°C)	Soften- ing point (°C) ^a	mp (°C)	Soften- ing point (°C)a
Quinol (IV)	360	380	455	8.71	260- 275	220	220	150
Resorcinol (V)	287- 298	230	415	11.49	247- 260	213	90- 110	80
3-Aminophenol (VI)	360	280	415	13.35	236- 250	185	150- 170	100
4-Aminophenol (VII)	360	440	470	8.12	290- 310	223	150- 170	06
2,2-Bis-(4-hydroxy- phenyl) propane (VIII)	360	290	420	20.17	208- 225	130	190- 210	155
Bis-(4-hydroxyphenyl) sulfone (IX)	360	300	420	12.97	215- 235	125	170- 185	130
4,4'-Dihydroxydiphenyl (X)	360	470	470	10.54	360	284	250- 270	180

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^aDetermined by the thermomechanical method.

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	Aci	d chlo ratio	ride	Intensive decomposition	Activation energy
Phenol	I	п	ш	(°C)	(kcal/ mol)
Resorcinol (IV)	60	-	40	350	38.39
3-Aminophenol (V)	60	-	40	350	28.30
	60	40	-	370	11.92
4-Aminophenol (VI)	60	-	40	325	7.25
2,2-Bis(4-hydroxyphenyl) propane (VIII)	90	-	10	400	16.41
	80	-	2 0	42 0	18.14
	60	-	40	400	19.62
Bis(4-hydroxyphenyl) sulfone (IX)	60	40	-	365	14.56
	60	-	40	360	18,52
4,4'-Dihydroxydiphenyl	60	40	-	360	13.79
(X)	60	-	40	355	11.98

TABLE 6b. Intensive Decomposition Temperatures and Activation Energies of Copolymers Synthesized from Different Bisphenols with a Mixture of Acid Chlorides I + II and I + III

determined that the decreasing stability order for the acid chlorides examined is in the order III > I > II, and for the bisphenols studied it is in the order X > IV > IX > VIII.

In agreement with the known concept that polyamides are relatively more stable than polyarylates [21-23], it has been also here found that the polymers obtained from terephthaloyl chloride (I) and 4aminophenol (VII) are thermally more stable than those derived from acid chloride I and quinol (IV).

The structure effects on the thermal stability of the polymers prepared were further emphasized by calculation of the thermal decomposition activation energies [25, 26] (Table 6b) by applying Doyle's equation [27]. The newly induced copolymer characteristics are very probably related to reduction of the intermolecular forces in the macromolecules of the copolymer. This is evidently due to random copolymerization leading to destruction of the molecular symmetry in homopolyarylates.

The fine structure of the synthesized polymers was further elucidated by a study of their IR spectra (Table 7). These polymers show a strong absorption band in the 1780-1760 cm⁻¹ region, attributable













	+ T cl	'erephtha hloride (I	loyl)	+ 4,4 for di	'-Bis-(ch mylmethe iphenyl (I	lloro- oxy) I)
Phenol	-NH-	-CO-	-0-		CO	-0-
Quinol (IV)		1720s	1100s		1730s	1100w
					1700s	
Resorcinol (V)		1740s	1100s		1780s	1100w
					1730s	
					1700s	
3-Aminophenol (VI)				3360b	1760s	1100w
					1 75 0s	
4-Aminophenol (VII)	3320ъ	1 72 0s	1100s	3360b	1760s	1100w
		1650s			1650s	
2,2-Bis(4-hydroxy-					1770s	
phenyl)propane (VIII	[)				1730s	1100s
Bis-(4-hydroxyphenyl	.)				1780s	1100w
sulfone (IX)					1650s	
4,4'-Dihydroxy-		1720	1100ss		1780s	1100w
diphenyl (X)					1730s	

TABLE 7. IR Spectra of Synthesized

 a b = broad, s = strong, ss = strong and splitted, w = weak.

to an ester carbonyl group. The strong band occurring at 1100 cm^{-1} in nearly all the compounds studied can be easily ascribed to an ether linkage. A strong band at 3500-3200 cm⁻¹ is evidently due to CH frequency.

EXPERIMENTAL

Infrared spectra were run on a Unicam S.P. 200. Melting points are uncorrected and were determined on a Kofler block. Terephthaloyl chloride (I) [27] was prepared by the action of

+ Bi forn pheny	ls-(4-ch nylmeth rl) sulfo	lloro- loxy- ne (III)	+ T ch	'erephtha loride <u>an</u>	loyl d II	+ T ch	Terephtha loride <u>ar</u>	aloyl Id III
NH	-CO-	-0	-NH-	CO	-0	-NH	-CO-	-0-
	1730s	1100w		1770s	1100s		1720s	1100s
	1700			17 2 0				
	1730s	1100w		1770s	1100s		1770s	1100ss
	1700s			1730s			1730s	
3350b	1760s	1100w	3350ь	1710s	1100s			
	1680s			1670s				
				1650s				
3320b	1760s		3450 _b	1730s	1100s	3450	1720s	1100s
	1740s		3350	1710s		3350	1640s	
	1680s			1660s				
				1770s	1100s			
				1730s				
				1725s	1100s			
	1760s	1100s		1770s	1100s		1770s	1100ss
				1730s			1720s	

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thionyl chloride on the corresponding acid. It crystallized from benzene in colorless needles, mp 81-83°C.

4,4'-Bis(carboxymethoxy) diphenyl [7] was prepared from 4,4'dihydroxydiphenyl and monochloroacetic acid in alkaline medium, and was crystallized from dioxane in needles, mp 262° C.

4,4'-Bis(chloroformylmethoxy) diphenyl (II) [7] was obtained by the action of thionyl chloride on the corresponding acid crystallized from n-hexane in rods, mp 78° C.

<u>Bis(4-carboxymethoxyphenyl) sulfone</u> [7] was prepared from bis(4-hydroxyphenyl) sulfone and monochloroacetic acid in alkaline medium crystallized from water in needles, mp $223-224^{\circ}$ C.

Bis(4-chloroformylmethoxyphenyl) sulfone (III) [7] was obtained by the action of thionyl chloride on the corresponding acid crystallized from an n-hexane-toluene mixture in needles, mp 108-109°C.

Experimental Procedure for Interfacial Polycondensation [5, 10, 11, 19]

The homopolyarylates and polyamidoarylates described in the present work were prepared as follows.

The calculated amount of acid chloride (0.0025 mol), dissolved in 7.5 mL of a dry water-immiscible organic solvent, was added all at once at room temperature to a vigorously stirred (2000-3000 rpm) aqueous solution of the phenolic component containing the equivalent amount of sodium hydroxide [5, 11]. After further stirring for 20 min, the organic solvent was steam distilled and the separated polymer was filtered and washed with distilled water until chloride free. After being again washed with a suitable organic solvent to remove the unreacted monomers, the purified polymer was dried to a constant weight in high vacuum at 50° C.

Experimental Procedure for Copolycondensation

The copolyarylates described hereafter were prepared by following the same technique previously adopted for the preparation of homopolyarylates. The only difference, however, was the use of 0.0025 mol of a mixture of two different acid chlorides dissolved in 7.5 mL of a dry water-immiscible organic solvent. The aminophenol, used at a concentration of 0.0025 mol, was treated with 0.0050 mol of sodium hydroxide dissolved in 15 mL of water.

Viscosity Measurements

The viscosities of a 0.5% solution of the polymer prepared—in a proper solvent—were measured at $25 \pm 0.01^{\circ}$ C using a Ubbelhode suspended level viscometer as modified by Ravikov [28, 29]. The flow times of pure solvents in the viscometers used throughout this work ranged from 80 to 120 s.

Thermomechanical Properties

The thermomechanical method [31, 32] was used for determining the temperature dependence of deformation caused by the action of a constant axial compression stress for a period of 10 s. The measurements were carried out using a stress of 2.5 kg/cm² by uniformly raising the temperature 1-2°C/min. The deformation percentage was calculated by applying the equation % deformation = (deformation obtained/sample thickness) imes 100

Thermogravimetric Analysis

This was carried out on a drivatograph instrument "Paulk-Paulk And Erdi, Budapest Hungaria". About 0.12-0.14 g of the polymeric substance was heated in carbon dioxide-free dry air. The temperature was raised from room temperature to 1000° C at a rate of 10° C/min.

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