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MACROMOLECULAR CHEMISTRY AND POLYMERIC MATERIALS

Acid-Catalyzed Oligomerization of Aromatic Ethers (Rolivsans) with Terminal Styrene and Methacrylate Groups

B. A. Zaitsev, G. I. Khromova, and I. D. Shvabskaya

Institute of Macromolecular Compounds, Russian Academy of Sciences, St. Petersburg, Russia

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Abstract—Cationic mechanism of transformation of unsaturated aromatic monomers contained in Rolivsans into thermosetting aromatic oligoethers with increased molecular weight was analyzed. The composition and chemical structure of Rolivsans as influenced by catalytic systems with various donor-acceptor properties were studied.

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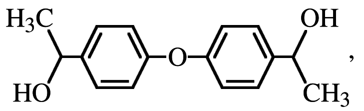
Rolivsans are low-viscosity thermosetting resins of new type, formed by reactions of secondary aromatic diols, such as di-[4-(1-hydroxyethyl)phenyl] ether (**I**)



with methacrylic acid catalyzed by strong acids [1–5]. Their thermal treatment yields heat-resistant glassy network polymers [6–10] which are used in (micro)-electronics and electrical engineering.

It has been found previously that Rolivsans contain the following monomer aromatic ethers: 4,4'–CH_2=CH–PhOPh–CH=CH_2 (M1), 4,4'–CH_2=CH–PhOPh–CH(CH_3)OCO–C(CH_3)=CH_2 (M2), 4,4'–CH_2=C(CH_3)–COO(CH_3)CH–PhOPh–CH(CH_3)OCO–C(CH_3)=CH_2 (M3), and oligoethers (OL) with terminal styrene-like and methacrylate groups.

The monomer-oligomer composition and properties of Rolivsans can be varied in a wide range depending on the synthesis conditions [3–5]. Low-viscosity compositions (500–1500 mPa s^{-1}) containing no solvents and predominantly consisting of monomers M1, M2, and M3 and small amounts (15–30%) of low-molecular-weight unsaturated oligomers (dimers and trimers) can be used as heat-resistant insulating casting and impregnating compounds.

In this study, we examined the formation of monomer–oligomer formulations with molecular weight of components increased due to higher content of aromatic oligoethers with terminal unsaturated groups. These formulations yield thermosetting polymers with controlled network density and can be used to prepare heat-resistant coatings and binders [11–12].

EXPERIMENTAL

The IR spectra were recorded on a Specord 75-IR device (thin films or $CHCl_3$ solutions). The exclusion liquid chromatography (ELC) of Rolivsans was performed on a GPC Waters chromatograph equipped with Ultrastyrogel (500 Å) columns at 20°C, using toluene as eluent. The number-average molecular weights of oligomers $\overline{M}_n$ were determined with a Hitachi–Perkin–Elmer 115 vapor osmometer. The sample unsaturation was studied on a ADS-4 ozonizer at 0°C (CCl_4 solvent). The viscosity and viscoelastic properties of Rolivsan MV-1 and of the products of its oligomerization were studied on a PIRSP-1 rheogoniometer with a cone-plate working unit (cone diameter 2 cm, cone angle 1°). The velocity gradients were varied from 2×10^{-2} to 4×10^{2} s^{-1}, and circular frequencies, from 2×10^{-2} to 10 s^{-1}.

Samples of Rolivsan MV-1 (series I and II) were prepared in accordance with TU (Technical Specifications) 6-36-57-0–91 by condensation of di-[4-(1-hydroxyethyl)phenyl] ether (**I**) with methacrylic acid in the presence of *p*-toluenesulfonic acid monohydrate (TSA-M) [3–5]; their compositions and properties are listed in Table 1.

Oligomer no. 1 was isolated from the initial reaction mixture (Rolivsan VM-1, series I) after its dilution with chloroform and subsequent precipitation

Table 1. Properties of Rolivsan MV-1*

Series	Composition, wt %				>C=C<, mol g^{-1}	$\overline{M}_n$	n_D^{20}	η^{25}, mPa s^{-1}	d^{20}, g cm^{-3}	τ^{140}, h
	M1	M2	M3	OL						
I	13	31	23	33	0.0058	400	1.585	1500	1.09	1.5
II	13	26	18	43	0.0056	420	1.590	1800	1.09	2.5

* >C=C< is the degree of unsaturation of oligomer, mole of double bonds per gram of product; $\overline{M}_n$, number-average molecular weight; and τ^{140}, time of gelation at 140°C.

with ethanol. The efficiency of oligomer isolation was monitored chromatographically (ELC) by the absence of peaks belonging to monomers M1, M2, and M3 in the product isolated. After a fourfold reprecipitation, the oligomer contained insignificant amounts (2–4%) of the initial monomers; the monomer-free product was obtained only after an eightfold reprecipitation. The properties of oligomer no. 1 were as follows: mp ~80°C, $\overline{M}_n$ ~ 1650, content of dimers with $\overline{M}_n$ ~500 (ELC data) 2–4%; found (%): C 79.6, H 6.5.

The degree of unsaturation of oligomer no. 1 was 0.37 mole of double bonds per 100 g of the product.

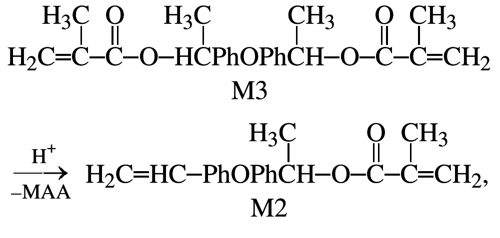
Two catalytic systems (CS A and CS B) were tested in oligomerization of Rolivsans catalyzed by TSA-M. Catalytic system A [solution of TSA-M (19.80 wt %) in polyphenyl ether (PPE, m-$C_{30}H_{22}O_4$, analytically pure grade, 5F 4E brand, mp 28°C] was prepared by through trituration of TSA-M (8.7841 g, 99% main compound) with liquid PPE (35.1365 g). Catalytic system B is a glycerol-like solution of TSA-M (23.63 wt %) in Cellosolve.

The acid-catalyzed oligomerization was performed by thorough mixing of Rolivsan with CS and storage of the homogenized composition at room temperature or at elevated temperature for a given time; then the composition and properties of the resulting compound were analyzed (Table 2).

To prepare oligomer nos. 2 and 3, CS A (1.05 g) was added with stirring to Rolivsan MV-1, series I (98.95 g), the homogenized composition was heated at 80°c for 1 h 35 min, and a sample was taken for isolation of OL no. 2. The process was continued for 2 h 40 min, and OL no. 3 was isolated from the reaction mixture. The product compositions were studied by ELC and IR spectroscopy. The composition of the sample containing OL no. 2 before isolation was as follows, wt %: 9 M1, 18 M2, 17 M3, and 56 OL. The sample was diluted with chloroform; the oligomer was precipitated with ethanol and dried at 20°C at reduced pressure. The precipitated product contained (wt %): 3 M1, 4 M2, 6 M3, and 87 OL no. 2, it is soluble in acetone and chloroform, $\overline{M}_n$ 1000. Oligomer no. 3 isolated by the similar procedure contained (wt %): 1 M1, 2 M2, 4 M3, and 93 OL; $\overline{M}_n$ 1220.

To prepare oligomer nos. 4 and 5, CS B (0.80 g) was added with stirring to Rolivsan MV-1, series I (99.20 g), and then one portion (50 g) of the homogenized composition was heated at 80°C for 2 h 10 min, and another portion (50 g) was stored at 20°C for 21 days. The compositions of the resulting products were analyzed as described above. The composition of the sample containing OL no. 4 before isolation was as follows, wt %: 3 M1, 6 M2, 4 M3, and 87 OL. No monomers were found in OL no. 4 after an eightfold reprecipitation; mp 90–110°C; found (%): C 79.60, H 6.70; 0.0030 mol of double bonds per gram of product, $\overline{M}_n$ 3375. The composition of the sample containing OL no. 5 before isolation was as follows, wt %: 2 M1, 5 M2, 3 M3, and 90 OL (no. 5). After precipitation and drying, we obtained oligomer no. 5 as a light brown resin with mp 95–115°C. After reprecipitation in the presence of hydroquinone (1%), the melting point of OL no. 5 increased to 125–135°C; found (%): C 81.20, H 6.70; 0.0037 mol of double bonds per gram of product, $\overline{M}_n$ 2700.

As shown previously, cleavage of esters (such as methacrylates) [1–5] and cationic addition polymerization of divinyl aromatic monomers with proton transfer [13–18] are of particular importance in catalyzed reactions of aromatic ethers containing secondary alcohol groups with carboxylic acids, such as methacrylic acid (MAA). The cleavage of dimethacrylate of aromatic diol (**I**), which is the main component of Rolivsan M3, and subsequent dimerization of the resulting vinyl aromatic monomer M2 are typical examples of such transformations [19]:

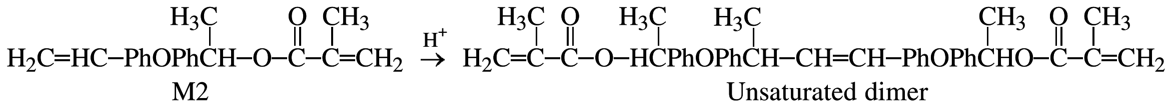


$$H_2C{=}\overset{H_3C}{\overset{|}{C}}-\overset{O}{\overset{\|}{C}}-O-H\overset{H_3C}{\overset{|}{C}}PhOPh\overset{CH_3}{\overset{|}{C}}H-O-\overset{O}{\overset{\|}{C}}-\overset{CH_3}{\overset{|}{C}}{=}CH_2$$

M3

$$\xrightarrow[-MAA]{H^+} H_2C{=}HC-PhOPh\overset{H_3C}{\overset{|}{C}}H-O-\overset{O}{\overset{\|}{C}}-\overset{CH_3}{\overset{|}{C}}{=}CH_2,$$

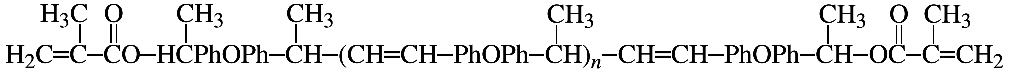
M2



$$H_2C{=}HC{-}PhOPh\overset{H_3C}{\overset{|}{C}}H{-}O{-}\overset{O}{\overset{\|}{C}}{-}\overset{CH_3}{\overset{|}{C}}{=}CH_2 \xrightarrow{H^+} H_2C{=}\overset{H_3C}{\overset{|}{C}}{-}\overset{O}{\overset{\|}{C}}{-}O{-}H\overset{H_3C}{\overset{|}{C}}PhOPh\overset{H_3C}{\overset{|}{C}}H{-}CH{=}CH{-}PhOPh\overset{CH_3}{\overset{|}{C}}HO{-}\overset{O}{\overset{\|}{C}}{-}\overset{CH_3}{\overset{|}{C}}{=}CH_2$$

M2 Unsaturated dimer

These data suggest that the intensified cleavage of methacrylates (M2, M3, and related compounds: dimers, trimers, etc.) and cationic addition polymerization of vinyl and divinyl aromatic compounds can yield linear unsaturated oligomers of the following type:



$$H_2C{=}\overset{H_3C}{\overset{|}{C}}{-}\overset{O}{\overset{\|}{C}}O{-}H\overset{CH_3}{\overset{|}{C}}PhOPh{-}\overset{CH_3}{\overset{|}{C}}H{-}(CH{=}CH{-}PhOPh{-}\overset{CH_3}{\overset{|}{C}}H)_n{-}CH{=}CH{-}PhOPh{-}\overset{CH_3}{\overset{|}{C}}H{-}O\overset{O}{\overset{\|}{C}}{-}\overset{CH_3}{\overset{|}{C}}{=}CH_2$$

Oligomerization was studied using two series of Rolivsans with different compositions and properties (Table 1). It should be noted that homogenization (dissolution) of TSA-M powder by direct stirring with a resin was rather difficult, because a high local concentration of the catalyst sharply accelerates oligomerization in situ and causes encapsulation of the TSA-M particles with the oligomer formed. To improve the homogenization conditions, we suggested two catalytic systems (CS A and CS B) based on PPE and Cellosolve solvents with different donor-acceptor properties.

The conditions of the acid-catalyzed process and the composition and characteristics of the oligomerized compositions and oligomers are listed in Table 2. The oligomers were isolated by dilution of the compositions with chloroform and subsequent precipitation with ethanol. Changes in the monomer-oligomer composition under different conditions of oligomerization of Rolivsans were studied by ELC (Fig. 1), IR spectroscopy (Figs. 2, 3), and rheokinetics (Fig. 4).

Gel chromatograms (Fig. 1) show that the content of the monomers decreases with time, with a simultaneous increase in the content of oligomers (especially, dimers and trimers) with number-average molecular weights of 500 – 800. Oligomerization was performed at a fixed content of catalysts both at 20 ± 2°C and at elevated temperatures. With the temperature increasing from 60 to 80°C, the process rate grows by more than a factor of 4, which corresponds to the known empirical van't Hoff rule.

It should be noted that the characteristics of unsaturation, content of carbon (Table 2), and ^{1}H NMR spectra are not fully consistent with the chemical structure of the oligomers, given above. The calculated degrees of unsaturation and carbon content are slightly higher than the experimental values. This is probably due to formation of small amounts of saturated structures (e.g., 1-methyl-3-phenylindan), along with the main structure of 1,3-diphenyl-1-butene (*trans*) type, in the course of acid-catalyzed dimerization of styrene. We also cannot rule out the side

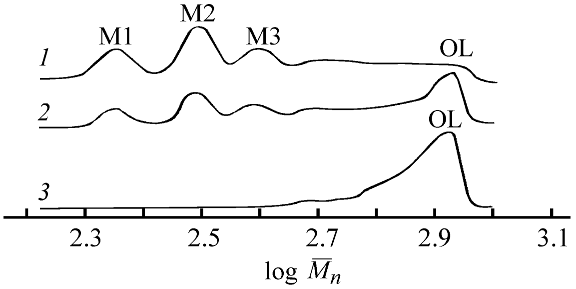


Fig. 1. Gel chromatograms of the (*1*) monomer–oligomer composition Rolivsan MV-1 (series I), (*2*) composition oligomerized by heating at 80°C for 1 h 35 min in the presence of CS A (content of TSA-M 1.19×10^{-2} M), and (*3*) oligomer no. 1 reprecipitated eight times to remove monomers. ($\overline{M}_n$) Molecular weight of oligomers.

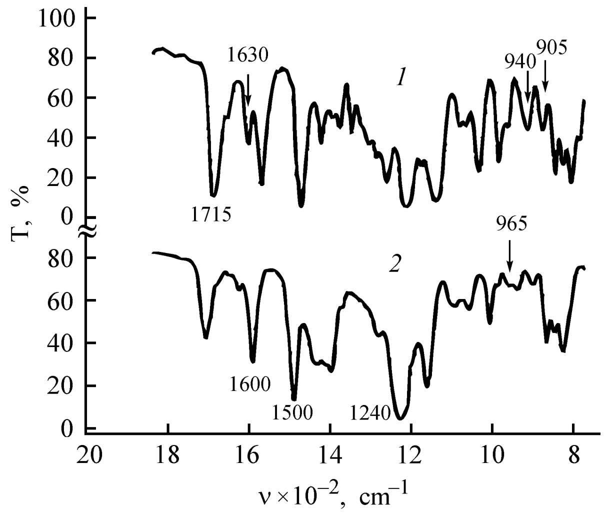


Fig. 2. IR spectra of the (*1*) monomer–oligomer composition Rolivsan MV-1 (series I) and (*2*) oligomer no. 3. (T) Transmission and (ν) wavenumber; the same for Fig. 3.

Table 2. Composition and properties of the reaction mixtures before and after oligomerization of Rolivsan MV-1 (series I)

Sample no.	Composition (oligomer)	CS	$c_{\text{TSA-M}} \times 10^2$, M	T, °C	τ, h	Composition, wt %				>C=C<, mol g^{-1}	Found, %		$\overline{M}_n$	mp, °C
						M1	M2	M3	OL		C	H		
1	Initial	–	–	–	–	13	31	23	33	0.0058	79.1	6.5	420	–
2	Oligomer no. 1*	–	–	–	–	1	2	6	91	–	–	–	–	–
3	Oligomer no. 1**	–	–	–	–	0	0	0	100	0.0037	79.6	6.5	1650	~80
4	Oligomerized	(A)	2.26	20	168	4	12	12	72	–	–	–	–	–
5	The same	(A)	2.26	80	1.2	4	8	7	81	–	–	–	–	–
6	"	(A)	1.19	80	1.6	9	18	17	56	–	–	–	–	–
7	Oligomer no. 2	(A)	1.19	80	1.6	3	4	6	87	–	–	–	1000	–
8	Oligomer no. 3	(A)	1.19	80	2.7	1	2	4	93	–	–	–	1220	–
9	Oligomerized	(B)	2.02	80	0.45	4	8	7	81	–	–	–	–	–
10	The same	(B)	2.02	60	1.2	4	11	9	76	–	–	–	–	–
11	"	(B)	1.08	20	504	3	6	4	87	–	–	–	–	–
12	Oligomer no. 4**	(B)	1.08	20	504	–	–	–	100	0.0030	79.6	6.7	3400	90–110
13	Oligomerized	(B)	1.08	80	2.2	2	5	3	90	–	–	–	–	–
14	Oligomer no. 5	(B)	1.08	80	2.2	–	–	–	–	0.0037	81.2	6.7	2700	125–135

* After fourfold reprecipitation; ** After eightfold reprecipitation.

reactions of oxidation on heating of unsaturated compounds in the presence of strong acids and of an addition of an aromatic solvent (toluene) and an inhibitor of the radical polymerization (hydroquinone) to the internal double bonds of the oligomer of Ar–CH(CH_3)–CH=CH–Ar type [16].

Similarly to the gel chromatograms, the IR spectra strongly change in the course of oligomerization. The initial IR spectrum of Rolivsan (Fig. 2, spectrum *1*) exhibits the absorption bands of the unsaturated and aromatic groups at 3080, 3060, 3030, 2975, 1630, 1600, 1500, 985, 965, 940, and 905 cm^{-1}, methyl and methylene groups at 2960, 2925, 2870, and 2850 cm^{-1}, methine group at 2900 cm^{-1}, and strong bands of the stretching vibrations of the carbonyl group in methacrylates at 1715 cm^{-1} and of aromatic ethers at 1240 cm^{-1} [4]. The IR spectrum of oligomer no. 3 (Fig. 2 spectrum *2*) isolated from the oligomerized composition (Table 2) shows the expected decrease in the intensity of the bands of the carbonyl groups at 1715 cm^{-1} and of out-of-plane bending vibrations H–C= at ~902 and 940 cm^{-1} belonging to the *p*–CH_2=CH–Ph–O–Ph and CH_2=C(CH_3)COO groups, respectively. Simultaneously, the band of H–C= out-of-plane bending vibrations of the –CH=CH– (*trans*) groups, whose concentration increases in the course of the stepwise dimerization reactions (cationic addition polymerization) of vinyl aromatic compounds [4, 19], grows in intensity.

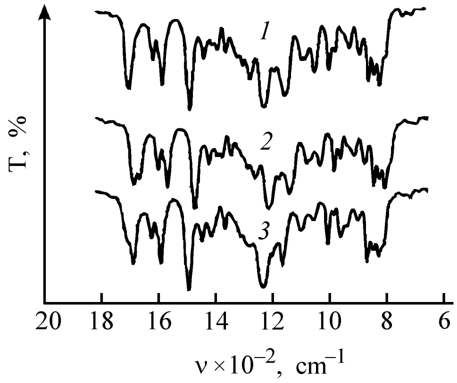


Fig. 3. IR spectra (20°C) of the monomer–oligomer composition Rolivsan MV-1 (series I) after its heating with CS B at a TSA-M content of 2.22 × 10^{-2} M. (*1*) 20°C (initial composition); (*2*) heated from 20 to 110°C over a period of 1 h and kept at 110°C for 2 h; (*3*) the same plus heating to 130°C over a period of 2 h.

Oligomerization was also performed at higher temperatures (110–180°C), at which the above reactions of addition polymerization are accompanied by cross-linking and cyclization. With heating, the intensity of the absorption bands of the terminal unsaturated groups at 1630, 940, and 905 cm^{-1} strongly decreases (Fig. 3, spectra *2*, *3*) owing to their consumption in copolymerization.

Another process of the cationic type, namely, cleavage of methacrylates to form vinyl aromatic compounds, methacrylic acid, and its copolymers, and also cationic addition polymerization of divinyl aromatic compounds occur simultaneously in the course of oligomerization. With heating, the band of the stretching vibrations of the carbonyl group >C=O of the methacrylic ester at 1715 cm^{-1} splits, and a new band of the MAA carboxy group appears at 1700–1685 cm^{-1} (Fig. 3, spectra *2*, *3*) [19].

Since the viscosity and gelation time (viability) are the most important process characteristics of the thermosetting plastics, which determine the possibility of their processing into a polymeric material, we studied the rheological properties of Rolivsans in the course of their catalytic oligomerization.

As can be seen from Fig. 4, each curve can be divided into two portions: that with comparatively slowly increasing viscosity, corresponding to the initial stage of the process, and that with rapidly increasing viscosity at the final stage of oligomerization (gelation). The intersection point of the tangents to these portions characterizes the gelation onset time τ_r. Table 3 we present the comparative data on τ_r and gelation time τ_g, determined visually from the loss of fluidity.

The fact that τ_g appreciably exceeds τ_r can be explained as follows: The rheokinetic method allows determination of the start of formation of 3*D* structures (microgel) from a sharp increase in the viscosity more precisely than does the visual determination of the fluidity loss observed at higher microgel concentrations.

Comparison of curves *2* and *4* (Fig. 4) shows that the rate of viscosity growth owing to oligomerization at 60°C is significantly higher in the experiment with addition of CS B (1.5 wt %), although the concentration of TSA-M (2.02×10^{-2} M) is lower in this case in this case than that in the experiment with addition of CS A (2 wt %) and TSA-M concentration of 2.26×10^{-2} M. The effect of the donor-acceptor complexes PPE–TSA–M and Cellosolve–TSA–M on the acidity of CS A and CS B and, thus, on their catalytic activity in the course of the Rolivsan oligomerization is illustrated in Fig. 5.

Table 3. Gelation time of Rolivsan MV-1 (series I) as influenced by the conditions of the acid-catalyzed oligomerization

CS	*C*, wt %	$c_{\text{TSA-M}} \times 10^2$, M	*T*, °C	τ_r	τ_g
				h	
–	0.00	0.00	140	1.3	1.5
A	1.00	1.14	80	2.5	3.5
A	2.00	2.28	80	0.75	1.0
A	3.30	3.76	80	0.5	0.7
A	2.00	2.28	60	3.0	>6.0
B	0.80	1.08	80	1.0	1.3
B	1.15	1.55	80	0.45	0.6
B	1.50	2.02	80	0.23	0.45
B	1.80	2.42	80	0.17	0.37
B	15.0	20.2	80	–	0.08
B	1.50	2.02	60	1.4	1.5
B	0.80	1.08	60	2.0	>5.0
B	1.50	2.02	20	–	72
B	15.0	20.2	20	–	>48

As can be seen, CS B containing Cellosolve is more reactive than CS A, other oligomerization conditions being equal. This is probably due to the higher basicity of Cellosolve as compared to PPE in which the lone electron pair of the ether oxygen is delocalized and interacts with the electron-acceptor phenyl groups, whereas in Cellosolve it is localized and interacts with the electron-donor alkyl groups. Thus, the electron-acceptor properties of PPE and Cellosolve interacting with TSA-M are strongly different. The electron density on the protonation centers in Cellosolve is higher owing to the lone electron pairs of the oxygen atoms.

At a CS A content decreased by a factor of 2, the rate of viscosity growth can be preserved by rais-

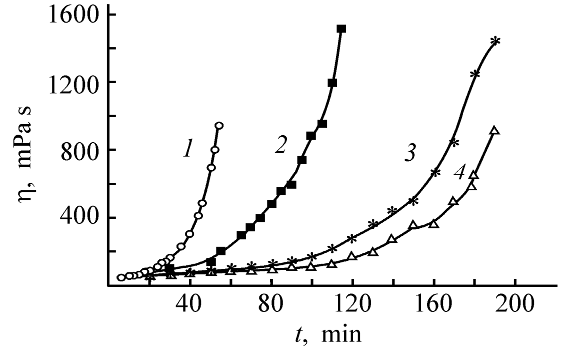


Fig. 4. Viscosity η of Rolivsan MV-1 (series I) vs. time *t* of oligomerization with addition of (*1*, *3*, *4*) CS A and (*2*) CS B. c_{CS}(wt %)/$c_{\text{TSA-M}}$, M: (*1*, *4*) 2/2.26, (*2*) 1.5/2.02, and (*3*) 1/1.14. Temperature, °C: (*1*, *3*) 80 and (*2*, *4*) 60.

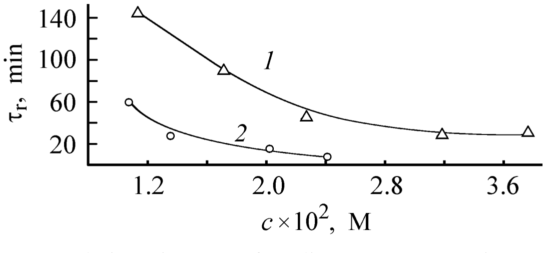


Fig. 5. Gelation time τ_r of Rolivsan MV-1 (series I) at 80°C vs. the TSA-M concentration *c*. Catalytic system: (*1*) A and (*2*) B.

ing temperature from 60 to 80°C (Fig. 3, curves *3*, *4*) which is also consistent with the van't Hoff rule.

CONCLUSIONS

(1) A procedure was developed for altering the composition and properties of Rolivsans by conversion of the monomer components [4,4'-divinyldiphenyl ether, 4-vinyl-4'-(1-methacryloyloxyethyl)diphenyl ether, and bis[4-(1-methacryloyloxyethyl)phenyl] ether] into linear unsaturated aromatic oligoethers with the terminal methacrylate groups .

(2) Under the conditions of the homogeneous catalytic oligomerization of Rolivsans at moderate temperatures (60–80°C), successive-parallel reactions of cleavage of methacrylates, formation of (di)vinylaromatic compounds, and addition polymerization of the latter with the proton transfer occur in 1–3 h.

(3) The catalytic systems with different donor-acceptor properties were developed, allowing variation of the rate Rolivsan oligomerization in a wide range.

(4) Low-melting oligoethers with the terminal methacrylate groups containing unsaturated and diphenyl oxide groups in the backbone were prepared and characterized.

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