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Cationic photopolymerization of carbazolyl- and phenothiazinyl-containing thiiranes

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Abstract

Photopolymerizations of ((9-carabzolyl)methyl)thiirane (CMT) and ((10-phenothiazinyl)methyl)thiirane (PMT) initiated with di-(*tert*-butylphenyl)iodonium tetrafluoroborate (BPIT), diphenyliodonium tetrafluoroborate (DPIT), cyclopropyldiphenylsulfonium tetrafluoroborate (CPS) and (η^5-2,4-cyclopentadien-1-yl) [1,2,3,4,5,6-η)-(1-methylethyl)benzene]-iron (+)-hexafluorophosphate(-1) (Irgacure 261) are reported. The influence of temperature and photoinitiator concentration on the rate of polymerization and conversion limit is discussed. The values of initiator exponent and activation energy for the photopolymerizations of CMT and PMT initiated with BPIT in 1,2-dichloroethane solution have been established. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Carbazole; Phenothiazine; Thiirane; Photopolymerization; Iodonium salt; Sulfonium salt; Iron arene complex

1. Introduction

Because of its advantages of high reactivity, a lack of oxygen inhibition and efficiency there is growing commercial interest in photoinduced cationic polymerization process. Diaryliodonium, triarylsulfonium salts and iron arene complex are technically useful cationic photoinitiators. Their chemistry has been studied in detail [1–3].

Carbazolyl-containing oligomers are known as a class of compounds with good photoconductivity and film-forming properties. These compounds have found use in the preparation of electrophotographic microfilms and photothermoplastic layers [4].

Carbazolyl-containing oligoethers are prepared by anionic [5] and cationic [6] polymerization of corresponding epoxy monomers. Like a number of other polymers with various aromatic amino groups poly(*N*-vinylphenothiazine) is also claimed to be useful in electrophotography [7].

Several studies [8,9] recently conducted at our laboratory have shown that carbazolyl-containing epoxides undergo facile photoinitiated cationic ring-opening polymerization in the presence of onium (iodonium, sulfonium) salts and iron arene complex.

It is known [10] that cyclic monomers containing one sulfur atom in the ring generally polymerize more readily, than their oxygen counterparts, over much larger range of temperatures. Cationic polymerization of thiiranes including (9-carbazolylmethyl) thiirane has been the subject of number of papers and review articles [11–13]. However, we have failed to find published data concerning the cationic photopolymerization of thiirane monomers.

With this in mind we have started the investigation of photoinduced cationic polymerization of ((9-carbazolyl)methyl) thiirane and ((10-phenothiazinyl)methyl)thiirane in the presence of onium salts and iron arene complex.

2. Experimental

2.1. Materials

((9-Carbazolyl)methyl)oxirane (CMO) ("Biolar", Latvia) was recrystallized twice from ethanol, m.p. 113 °C. Phenothiazine (PT) was used as received from Aldrich.

2.2. ((9-Carabzolyl)methyl)thiirane (CMT)

Potassium thiocyanate (20 g, 0.2 mol) and 10 ml of water were added to 22.3 g (0.1 mol) of CMO dissolved in 100 ml of 1,4-dioxane. The reaction mixture was stirred for 36 h at room temperature. Then the organic layer was separated, 20 g (0.2 mol) of potassium thiocyanate, 10 ml of water were added and the reaction mixture was stirred for 8 h. Thin layer chromatography (Silufol UV-254 plates and benzene as an eluent were used) after this time showed the end of the

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reaction. The mixture was extracted with toluene. The organic layer was washed with water, dried over anhydrous magnesium sulfate, treated with activated charcoal and filtered. After removal of toluene, the residue was washed with ethanol. 17.6 g (73.6%) of CMT was obtained. The monomer was recrystallized from ethanol (m.p. 100–101 °C).

^{1}H NMR ($CDCl_3$, δ, ppm): 2.31 (2H, m, CH_2S), 3.16 (1H, m, CH), 4.38 (2H, AB part of ABX system, N–CH_2; $J_{AB} = 15.5$, $J_{AX(trans)} = 6.5$, $J_{BX(cis)} = 5.5$ Hz), 7.34 (5H, m, CH_{Ht}), 8.05 (3H, m, CH_{Ht}).

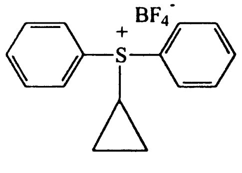
Elemental analysis for $C_{15}H_{13}NS$ (239.34): calculated: C 75.28%; H 5.48%; N 5.85%; S 13.40%; found: C 74.88%; H 5.58%; N 6.10%; S 13.21%.

UV (λ_{max}): 250, 290, 330, 340 nm.

2.3. ((10-Phenothiazinyl)methyl)oxirane (PMO)

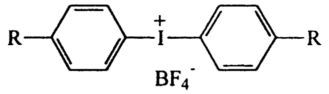
A mixture of 19.9 g (0.1 mol) of PT, 138.8 g (1.5 mol) of 3-chloro-1,2-epoxypropane, 16.8 g (0.3 mol) of powdered potassium hydroxide and 11.2 g (0.2 mol) of calcium oxide was stirred for 5 h at 35–40 °C temperature. At the end of the reaction (it was determined by thin layer chromatography using Silufol UV-254 plates and chloroform as an eluent) the mixture was extracted with diethyl ether. The organic layer was washed with water (until neutral water was obtained), dried with anhydrous magnesium sulfate, treated with activated charcoal, and filtered. After removal of diethyl ether and the excess of 3-chloro-1,2-epoxypropane, the fractional vacuum distillation of crude product gave 15.81 g (yield 62.0%) of PMO with a b.p. 216–218 °C at 2 mm Hg.

^{1}H NMR ($CDCl_3$, δ, ppm): 2.67 (2H, AB part of ABX system, CH_2O; $J_{AB} = 5$, $J_{AX(trans)} = 4$, $J_{BX(cis)} = 3$ Hz), 3.11 (1H, m, CH), 3.84 (2H, AB part of ABX system, N–CH_2; $J_{AB} = 16$, $J_{X(trans)} = 5$, $J_{BX(cis)} = 3$ Hz), 6.92 (8H, m, CH_{Ht}).



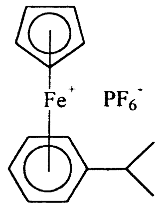
Sulfonium salt

(CPS)



Iodonium salts

(BPIT and DPIT)



Iron arene complex

Irgacure 261

Elemental analysis for $C_{15}H_{13}NOS$ (255.34): calculated: C 70.56%; H 5.13%; N 5.48%; S 12.56%; found: C 70.39%; H 5.24%; N 5.75%; S 12.7%.

2.4. ((10-Phenothiazinyl)methyl)thiirane (PMT)

Potassium thiocyanate (19.4 g, 0.2 mol) and 10 ml of water were added to 15.4 g (0.06 mol) of PMO dissolved in 100 ml of 1,4-dioxane. The mixture was stirred for 15 h at room temperature, then extracted with diethyl ether and washed with water. Diethyl ether was removed and the residue was dissolved in 1,4-dioxane (~100 ml). A new portion of potassium thiocyanate (19.4 g) and water (10 ml) were added to the reaction mixture and stirred. Thin layer chromatography of the mixture revealed the disappearance of starting material after 10 h. Then the mixture was extracted with diethyl ether and washed with water. After removal of diethyl ether, the resulting monomer was isolated by column chromatography. A column applied was packed with aluminum oxide (Brockmann II, neutral). A system of diethyl ether–petroleum ether (3:4) was used as an eluent. Yield of PMT was 10.7 g (65.9%). The monomer obtained was recrystallized from ethyl alcohol, m.p. 120–121 °C.

^{1}H NMR ($CDCl_3$, δ, ppm): 2.46 (2H, AB part of ABX system, CH_2S; $J_{AB} = 6.4$, $J_{AX(trans)} = 1.5$, $J_{BX(cis)} = 1.3$ Hz), 3.28 (1H, m, CH), 4.14 (2H, AB part of ABX system, N–CH_2; $J_{AB} = 15.5$, $J_{AX(trans)} = 7.7$, $J_{BX(cis)} = 3.0$ Hz), 6.80–7.25 (8H, m, CH_{Ht}).

Elemental analysis for $C_{15}H_{13}NS_2$ (271.41): calculated: C 66.38%; H 4.83%; N 5.16%; S 23.63%; found: C 66.15%; H 4.85%; N 5.20%; S 23.29%.

UV (λ_{max}): 260, 290, 310 nm.

Photoinitiators: di-(*tert*-butylphenyl)iodonium tetrafluoroborate $[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-$ and diphenyliodonium tetrafluoroborate $(C_6H_5)_2I^+BF_4^-$ were synthesized and purified as described before [14]. Cyclopropyldiphenylsulfonium tetrafluoroborate $C_3H_5S^+(C_6H_5)_2BF_4^-$ and (η^5-2,4-cyclopentadien-1-yl) [1,2,3,4,5,6-η)-(1-methylethyl) benzene]-iron(+)-hexafluorophosphate(-1) (Irgacure 261) were used as received from Aldrich. $BF_3{\cdot}O(C_2H_5)_2$ was refluxed and distilled over CaH_2, b.p. 125–126 °C.

Tetrahydrofuran (THF) ("Lachema") was refluxed over sodium for 40 h and distilled. 1,4-Dioxan was treated (2 h) with sodium, and then distilled. 1,2-Dichloroethane was distilled before use. The solvents, hexane ("POCH"), diethyl ether ("Lachema"), chloroform ("Lachema") and isopropyl alcohol ("Reachim") were used as received.

2.5. Polymerization

The photopolymerizations of the monomers were carried out in a quartz tube in the presence of air in

1,2-dichloroethane solution containing the initiator at different temperatures. The UV light source was a 240 W medium pressure mercury lamp (Model DRT-240, Russia) set at a distance of 6 cm. The light intensity on the sample was about 15 mW cm^{-2} (measured with a Neo-Lab UV measuring units). Conversion of the monomer, number average molecular weight, polydispersity index and number average degree of polymerization of the obtained oligomers were measured by GPC after irradiation of the samples for various periods of time.

The cationic polymerization of CMT initiated with $BF_3{\cdot}O(C_2H_5)_2$ was carried out in 1,2-dichloroethane solution ($[CMT]_0 = 0.5$ M) at 40 °C temperature for 3 h. Aqua ammonia was added to the reaction mixture for neutralization of the initiator. The obtained product was isolated by precipitation into isopropyl alcohol.

The anionic polymerization of CMT was initiated with butyl lithium, which was introduced in to reaction mixture frozen to −78 °C. The polymerization of CMT was carried out in tetrahydrofuran at room temperature for 24 h under the nitrogen atmosphere. The reaction was stopped by allyl bromide. The product obtained was isolated by precipitation into isopropyl alcohol.

2.6. Measurements

Gel permeation chromatography (GPC) was carried out on the modified liquid chromatograph Milichrom (Chernogolovka, Russia) using a special column packed with “Lichrispher Si 100” (1,4-dioxan was used as an eluent at flow rate 200 μl/min) or Water Styragel, Pumpe Waters 590 chromatograph (eluent-tetrahydrofuran). All data are referenced to narrow polystyrene standards.

Glass transition temperature (T_g) of the resulted oligomers was determined using Perkin-Elmer DCS-7 apparatus. Samples of 5–10 mg in solid form were put in aluminum pans and heated at a scan rate of 10 °C/min under a nitrogen flow. The temperature scale of the DSC was calibrated from the melting points (m.p.) of high purified metals such as indium (m.p. 429.78 K) and zinc (m.p. 692.65 K).

^{1}H NMR spectra were obtained on TESLA BS-487 (80 MHz) and JOEL FX 100 (100 Hz) spectrometers with $CDCl_3$ as the solvent, the chemical shifts are expressed in parts per million downfield to hexamethyldisiloxane (HMDS) used as internal standard.

3. Results and discussion

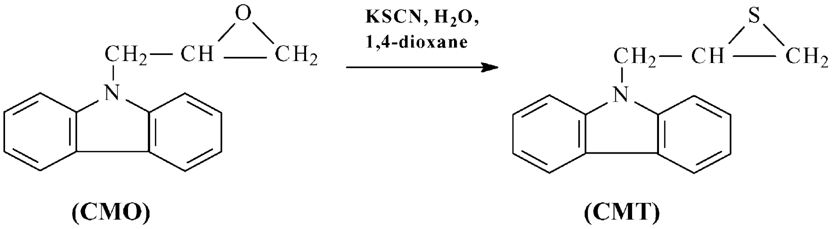
3.1. Synthesis

CMT was prepared by the reaction of CMO with potassium thiocyanate in the presence of water in 1,4-dioxane solution (Scheme 1). PMT was synthesized [15] by the reaction of 3-chloro-1,2-epoxypropane with phenothiazine in the presence of potassium hydroxide and calcium oxide, followed by the reaction of the product obtained (PMO) with potassium thiocyanate (Scheme 2).

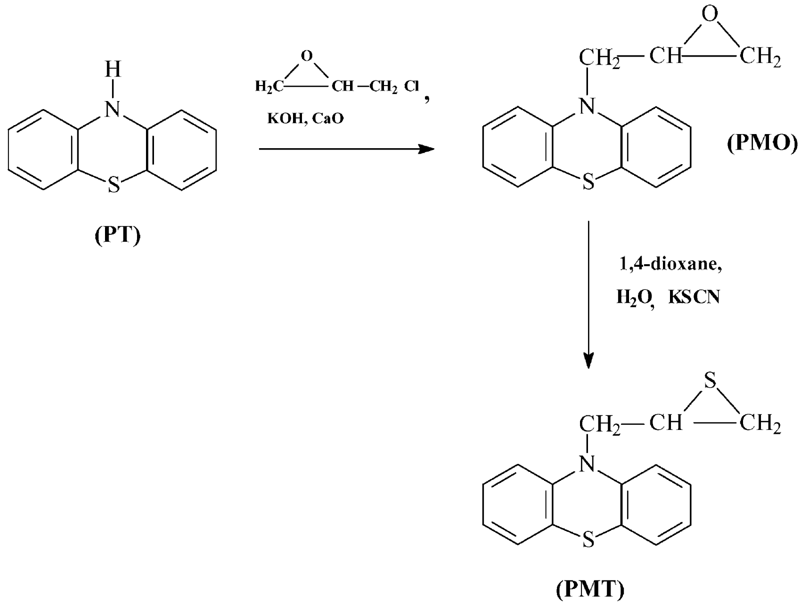
3.2. Photopolymerization

The cationic photopolymerizations of CMT initiated with different initiators, iodonium, sulfonium salts and iron arene complex at 30 °C have been investigated (Fig. 1 curves 1–3 and Fig. 2, curve 2). The highest initial rate is a characteristic of CMT photopolymerization initiated with DPIT (photopolymerization rate $R_p = 11.3 \times 10^{-4}$ mol/l s), however, in this case the termination of the reaction occurs at 80% limit conversion (Fig. 1, curve 1). A lower initial rate and a higher degree of conversion (ca. 90%) is a characteristic of CMT photopolymerizations initiated with BPIT and CPS (Fig. 1, curve 2 and Fig. 2, curve 2). When Irgacure 261 is used as photoinitiator, the rate of CMT photopolymerization in the initial period is high ($R_p = 4.62 \times 10^{-4}$ mol/l s), however, after some time the photopolymerization slows down considerably and finally only 45% conversion is reached after 90 min irradiation (Fig. 1, curve 3). It can be concluded from the data presented that the efficiency of DPIT, BPIT and CPS is much higher in the cationic photopolymerization of CMT than that of Irgacure 261. It is evident that nature of cation of the initiators influences the results of CMT photopolymerization.

For the comparison kinetic curve of CMO photopolymerization conducted at 30 °C is presented in Fig. 2. It is evident that rates of CMT and CMO photopolymerizations initiated with BPIT are similar at the very initial stage of the reactions. However, after 5 min irradiation CMO photopolymerization slows down and only 42% conversion of the monomer is reached after 60 min exposure. On the contrary, the rate of CMT photopolymerization remains very high and 90% conversion is achieved after 20 min of irradiation. The higher values of molecular weight and the



Scheme 1.



Scheme 2.

polydispersity index (PD) were observed for CMT oligomers. The CMT oligomers of degree of polymerization (DP) of 13 are obtained, while DP of CMO oligomers is ca. 7. It can be assumed from the data presented above that the reactivity of CMT is much higher in cationic photopolymerization than that of CMO.

In order to established the activation energy value the photopolymerization of CMT with BPIT was conducted at various temperatures (Fig. 2, curves 2–4). It must be noted that the increase in the temperature increases the initial rate of CMT photopolymerization and the degree of the monomer conversion.

After measuring the rate constants the plot of $\ln k$ versus $1/T$ was obtained for CMT photopolymerization with BPIT at various temperatures (Fig. 3). The activation energy of 82.9 kJ/mol for CMT photopolymerization was established.

Conversions versus time curves for the photopolymerization of CMT initiated with varying amount of BPIT are presented in Fig. 4. It must be noted that the induction period (ca. 2 min) is a characteristic of CMT photopolymerization initiated with 0.3 mol% of BPIT. It is evident that the increase in the initiator concentration leads to the increase of initial rate of the photopolymerization and degree of the

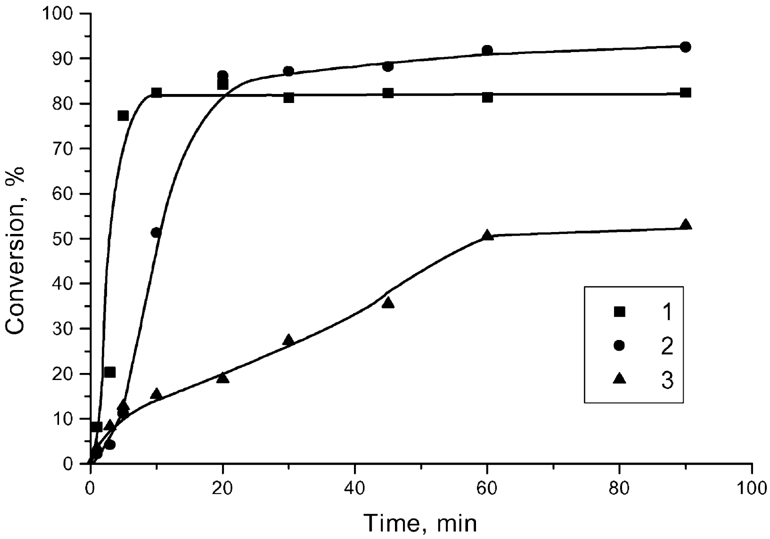


Fig. 1. Conversion versus times curves for the photopolymerization of CMT, initiated with: (1) DPIT; (2) CPS; (3) Irgacure 261 at 30 °C. $[CMT]_0 = 1$ mol/l. Initial concentration of initiator 1 mol% per monomer.

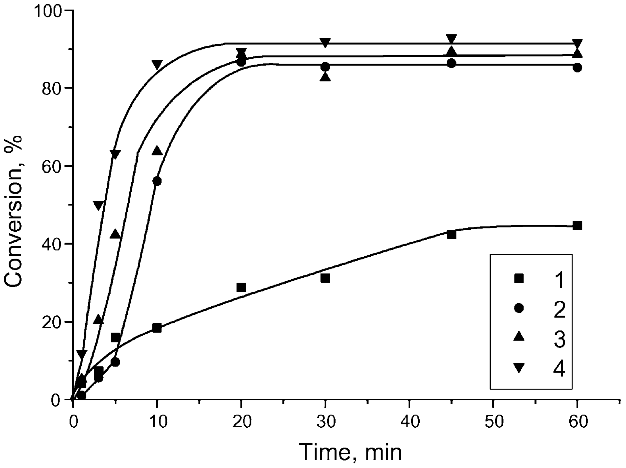


Fig. 2. Conversion versus times curves for the photopolymerization of CMO at 30 °C (1) and of CMT: (2) −30 °C; (3) −40 °C; (4) −50 °C. Initiator BPIT (1 mol% per monomer). $[CMT]_0 = [CMO]_0 = 1$ mol/l.

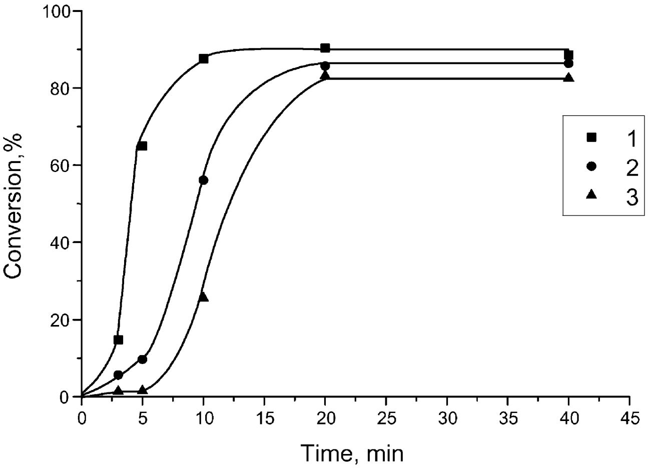


Fig. 4. Conversion versus time curves for the photopolymerization of CMT initiated with BPIT at 30 °C. Initial concentration of BPIT (mol% per monomer): (1) −3; (2) −1; (3) −0.3. $[CMT]_0 = 1$ mol/l.

conversion. The value of the initiator exponent was calculated using these data. The BPIT exponent of 1.04 was established.

In order to compare the characteristics of poly(CMT) obtained using different methods of polymerization, dark cationic and anionic polymerizations of the monomer were carried out. Fig. 5 presents gel permeation chromatograms of poly(CMT) obtained by cationic, anionic polymerization and cationic photopolymerization reactions. It is evident that in the case of the cationic polymerization the product is rich in the low-molar mass fraction and only small amount of high-molar mass fraction is observed. On the contrary, the product of anionic polymerization of CMT is rich in the high-molar mass fraction. Only small amount of low-molar mass fraction is observed as the product of anionic polymerization.

The existence of the low-molar mass fraction in poly(CMT) obtained by the cationic polymerization and cationic photopolymerization can be explained by the cyclization reactions of CMT. It is known [16,17] that cationic polymerization of various thiiranes is accompanied by degradation into cyclic oligomers. It was shown [10] that iodonium and sulfonium salts initiate the radiation-induced degradation of polythiiranes, which leads to the formation of various cyclic oligomers.

Table 1 summarizes the characteristics of poly(CMT) obtained using different methods of polymerization. It is evident that a high value of PD is a characteristic of poly(CMT) prepared by all the methods. Such a high value of PD indicates the presence of chain transfer reactions. Cationic photopolymerization of CMT leads to the

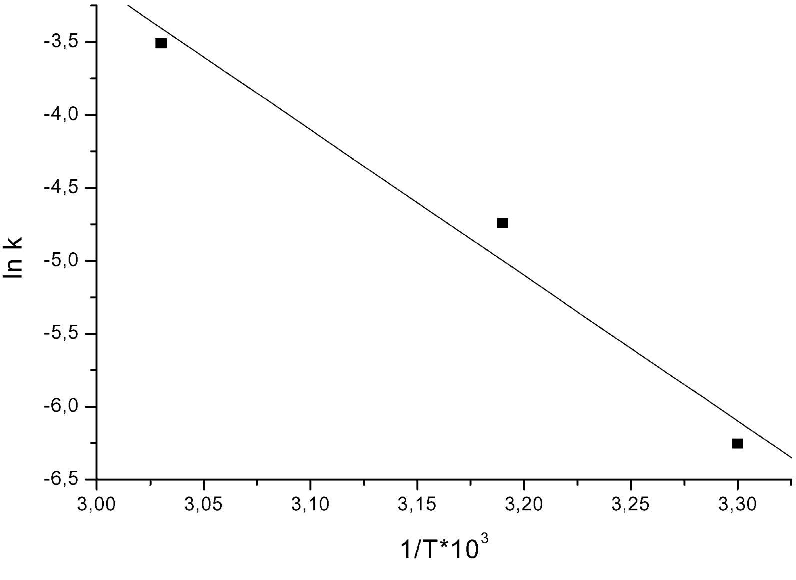


Fig. 3. ln k versus $1/T$ plot for the photopolymerization of CMT. $[CMT]_0 = 1$ mol/l. Initiator BPIT (1 mol% per monomer).

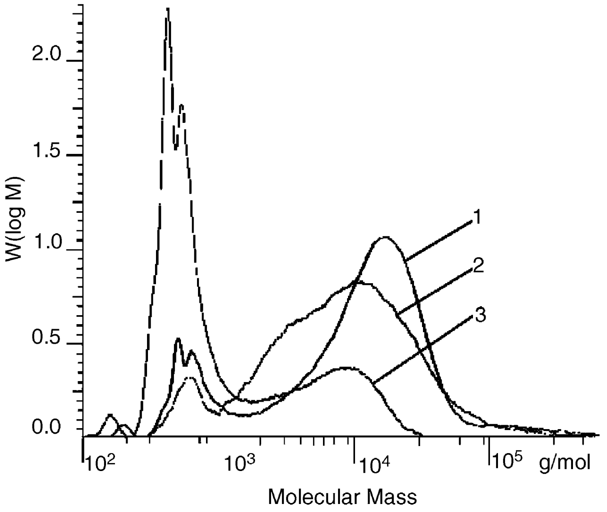


Fig. 5. Distribution of molecular mass of poly(CMT) obtained by different methods: (1) cationic photopolymerization, (2) anionic polymerization, (3) cationic polymerization.

oligomers of much higher molecular weight compared to those obtained by cationic polymerization. The oligomers of DP 13 are obtained in the cationic photopolymerization reaction, while DP of CMT oligomers is ca. 4 for the cationic polymerization.

Fig. 6 shows the results of cationic photopolymerization of PMT initiated with iodonium salts and iron arene complex in 1,2-dichloroethane solution. The highest initial rate and complete termination of the reaction after reaching limit conversion of ca. 50% is a characteristic of PMT photopolymerization initiated with Irgacure 261. PMT photopolymerizations with the iodonium salts exhibit very similar limit conversion (50–55%) to that with Irgacure 261. When iodonium salts are used as photoinitiator, an induction period, which lasts 5–10 min, is observed. Thus the initial rate of the photopolymerization depends on the nature of photoinitiator, however it does not influence the limit degree of conversion.

The results listed in Table 2 demonstrate that the photopolymerization of PMT initiated with BPIT gives oligomers of highest DP (ca. 7) and PD. Meanwhile oligomers of DP of 5 are obtained in the photopolymerization of PMT with Irgacure 261.

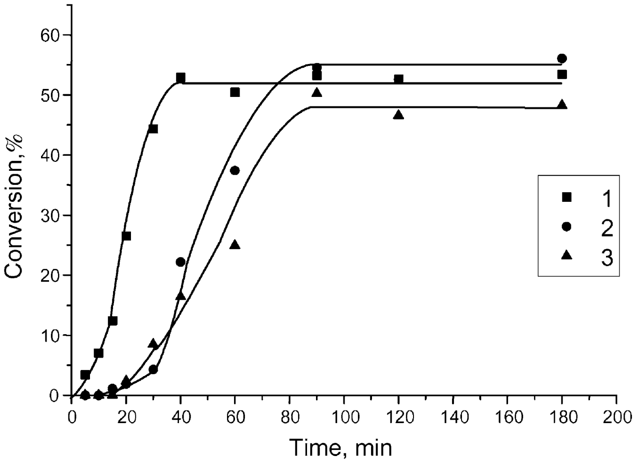


Fig. 6. Conversion versus time curves for the photopolymerization of PMT, initiated with: (1) Irgacure 261, (2) $[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-$, (3) $(C_6H_5)_2I^+BF_4^-$ at 30 °C. $[[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-]_0 = [(C_6H_5)_2I^+BF_4^-]_0 = [\text{Irgacure 261}]_0 = 1$ mol% per monomer. $[PMT]_0 = 0.5$ mol/l.

Table 2

Characteristics of the oligomers obtained on the photopolymerization of PMT initiated with different initiators at 30 °C[a]

Initiator	Irradiation time (min)	$\bar{M}_n$	$\bar{M}_w$	$\bar{M}_w/\bar{M}_n$	$\overline{DP}$[b]
$[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-$	120	1861	2724	1.46	7
$(C_6H_5)_2I^+BF_4^-$		1798	2467	1.37	6
Irgacure 261		1349	1879	1.39	5

[a] $[PMT]_0 = 0.5$ mol/l, $[[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-]_0 = [(C_6H_5)_2I^+BF_4^-]_0 = [\text{Irgacure 261}]_0 = 1$ mol/% per monomer.

[b] Number average degree of polymerization measured by GPC.

In order to established the influence of initiator concentration on the reaction rate the PMT photopolymerizations initiated with various amounts of BPIT were carried out (Fig. 7). The PMT photopolymerization initiated with 1 mol% of BPIT starts after induction period, which lasts ca. 10 min. The increase in concentration of the initiator increases the initial rate of PMT photopolymerization with BPIT, however finally very similar limit conversion of ca. 60% is reached in all cases. The BPIT exponent of 0.38 for the PMT photopolymerization was found.

The effect of the temperature on the PMT photopolymerization initiated with BPIT is shown in Fig. 8. It should

Table 1

Characteristics of the CMT oligomers[a]

Monomer	Initiator	$\bar{M}_n$	$\bar{M}_w$	$\bar{M}_w/\bar{M}_n$	$\overline{DP}$[b]	T_g (°C)
CMT	$[n(CH_3)_3CC_6H_4]_2I^+BF_4^-$	3103	27340	8.81	13	101.9
	$CH_3CH_2CH_2CH_2Li$	5850	25690	4.39	24	114.4
	$BF_3{\cdot}O(C_2H_5)_2$	943	3745	3.97	4	84.1

[a] Photopolymerization of CMT: $[CMT]_0 = 1$ mol/l, initiator BPIT (1 mol% per monomer) at 30 °C temperature. Cationic polymerization of CMT: $[CMT] = 0.5$ mol/l, initiator $BF_3{\cdot}O(C_2H_5)_2$ (1 mol% per monomer) at 40 °C. Anionic polymerization of CMT: $[CMT]_0 = 0.04$ mol/l, initiator $CH_3CH_2CH_2CH_2Li$ at room temperature.

[b] Number average degree of polymerization measured by GPC.

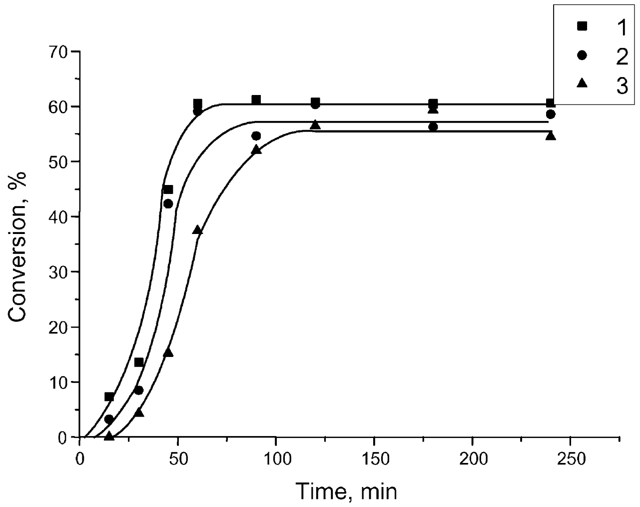


Fig. 7. Conversion versus time curves for the photopolymerization of PMT initiated with $[n\text{-}(CH_3)_3CC_6H_4]_2I^+BF_4^-$ at 30 °C. Initial concentration of photoinitiator (mol% per monomer): (1) −5; (2) −3; (3) −1. $[PMT]_0 = 0.5$ mol/l.

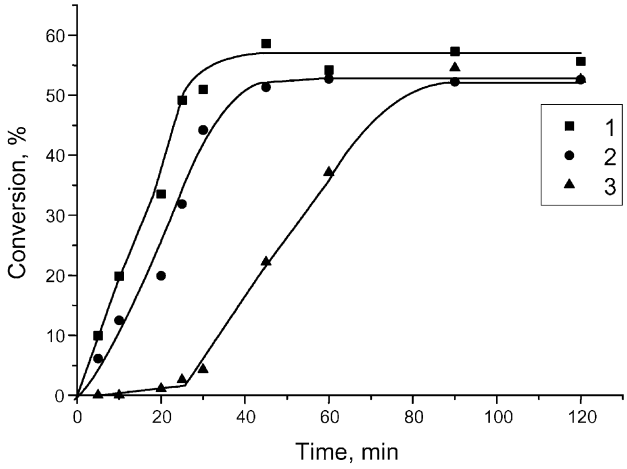


Fig. 8. Conversion versus time curves for the photopolymerization of PMT initiated with BPIT (1 mol% per monomer) at: (1) 50 °C, (2) 40 °C, (3) 30 °C. $[PMT]_0 = 0.5$ mol/l.

be noted that increase in the temperature significantly enhances the rate of PMT photopolymerization with BPIT. A limit conversion increases with increasing temperature. 30 °C temperature results 50% limit conversion of the reaction. Meanwhile ca. 60% limit conversion is achieved when the PMT photopolymerization with BPIT is conducted at 50 °C. The activation energy of 99.73 kJ/mol was found from the plot $\ln k$ versus $1/T$ for the photopolymerization of PMT with BPIT. It should be noted that the activation energy values established for the CMT and PMT photopolymerization do not contradict with experimental results of other authors. For example, Sigwalt and Spassky [13] found the activation energy of 75 kJ/mol for the polymerization of propylene sulfide in the presence of $BF_3{\cdot}OEt_2$.

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