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MACROMOLECULAR COMPOUNDS AND POLYMERIC MATERIALS

Synthesis of Photosensitive Cyclopropane-Containing Polymers

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Abstract—New cyclopropyl methacrylate monomers were prepared, their radical polymerization was performed, and the composition and structure of the polymers obtained, containing reactive UV-sensitive fragments, were determined. Experiments on photochemical cross-linking revealed photosensitivity of the polymers synthesized. The materials based on them are optically transparent and can be used in microelectronics and optics.

Keywords: cyclopropane, photosensitivity, optical transparency, phenyl-substituted cyclopropyl methacrylate

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Macromolecules containing various photoreactive fragments in macrochains exhibit a valuable set of properties. Such polymers can be cross-linked under the action of radiation, which allows preparation of resists for microelectronics. Therefore, preparation of new types of photosensitive polymers for microelectronics attracts much attention of researchers [1–5].

Polymers containing cyclopropane units with various functional substituents in macromolecules are of much interest, because they exhibit a balanced set of properties, including photosensitivity and optical transparency, and can be used in polymer resists and optically transparent materials in microelectronics and optics. One of the ways of preparing polymers containing cyclopropane units with reactive functional groups is polymerization of new monomers [6–9].

In this study, we prepared new cyclopropane-containing methacrylate monomers, analyzed the relationships of their radical polymerization, and determined the properties of the polymer samples obtained.

We suggested a new strategy of the synthesis of functionally substituted polymethacrylate with pendant functional cyclopropane fragments, capable of forming photosensitive elastic films. We also studied the structure and properties of the polymers with the aim of developing new photosensitive and transparent polymers.

EXPERIMENTAL

The IR spectra of the polymer films were determined with an AgilentCary 630 FTIR device, and the ^{1}H NMR spectra, with a Bruker AFR-300 spectrometer in $CDCl_3$; the chemical shifts were determined relative to tetramethylsilane. The purity of the compounds synthesized was checked by gas–liquid (GLC) and thin-layer (TLC) chromatography. The GLC analysis was performed with LKhM-8 MD (model 3) and Tsvet-100 chromatographs. Apiezon-L (15%), XE-60 (5%), and poly(ethylene glycol adipate) (PEGA) (5%) were used as liquid phases. The supports were Chromaton and Celite-547; the carrier gas was helium. TLC analysis was performed using glass plates of sizes 60 × 130, 130 × 180, and 230 × 300 mm with 1–2 mm thick unfixed SiO_2 layer and Silufol UV plates of sizes 30 × 60 and 160 × 160 mm with the fixed adsorbent layer.

The purity of all the starting compounds was 98–99.9%. The starting compounds had the following characteristics: styrene (Acros Organics), bp 145°C, n_D^{20} 1.5462, d_4^{20} 0.9090; *p*-chlorostyrene (Alfa Aesar), bp 191–192°C, n_D^{20} 1.5650, $d_4{}^{20}$ 1.0868; *p*-nitrostyrene (Alfa Aesar), bp 93–96°C, n_D^{20} 1.5753, d_4^{20} 1.1632; ethyl diazoacetate (synthesized by us), bp 140–141°C, n_D^{20} 1.4610, d_4^{20} 1.085; methacryloyl chloride (synthesized by us), bp 95–96°C, n_D^{20} 1.4420, d_4^{20} 1.070.

Reaction of ethoxycarbonylcarbene with phenyl-substituted styrenes. A mixture of 0.6 mol of styrene or *p*-phenyl-substituted styrene and 0.2 g of anhydrous copper sulfate (catalyst) was heated to 40–50°C, and 0.5 mol of ethyl diazoacetate was added dropwise with stirring at a rate of 10–12 g h^{-1}. After the whole amount of ethyl diazoacetate was added and the nitrogen evolution ceased, the mixture was cooled to room temperature. The catalyst ($CuSO_4$) was filtered off and extracted several times with diethyl ether, and the organic phases were combined. Then, the ether was distilled off, and the residue was distilled in a vacuum. The first fraction contained excess styrene or phenyl-substituted styrene, and then the main fraction (products 1–3) was distilled.

The IR spectra of 1–3 contain absorption bands at 3095–3100, 1030–1035 ($\nu_{\text{C–H ring}}$); 2980, 2940, 2880 ($\nu_{\text{C–H aliph}}$), 750–760 ($\nu_{\text{C–Cl}}$); 1690, 1720 ($\nu_{\text{C=O}}$); 1450–1500; 1570–1620, 1335, and 1525 cm^{-1} (NO_2, for 3); and out-of-plane C–H bending vibration bands of monosubstituted benzene at 690–750 cm^{-1} for 1.

2-Phenyl-1-ethoxycarbonylcyclopropane (1). Yield 90%, bp 68–70°C (3 mm Hg), n_D^{20} 1.5125, d_4^{20} 1.08. ^{1}H NMR spectrum, δ, ppm: 6.95 m ($5H_{\text{arom}}$), 1.92 m (1H, $CH_{\text{cyclopropane}}$), 2.56 m (1H, $CH_{\text{cyclopropane}}$), 1.65 m (1H, $CH_{2\text{cyclopropane}}$), 1.62 m (1H, $CH_{2\text{cyclopropane}}$), 3.85 q (2H, CH_2), 0.96 t (3H, CH_3). Found, %: C 75.20, H 7.91. $C_{12}H_{14}O_2$. Calculated, %: C 75.79, H 7.37.

*2-p-Chlorophenyl-1-ethoxycarbonylcyclopropane (**2**).* Yield 82%, bp 85–87°C (3 mm Hg), n_D^{20} 1.5640, d_4^{20} 1.32. ^{1}H NMR spectrum, δ, ppm: 7.10 m ($4H_{\text{arom}}$), 2.08 m (1H, $CH_{\text{cyclopropane}}$), 2.55 m (1H, $CH_{\text{cyclopropane}}$), 1.71 m (1H, $CH_{2\text{cyclopropane}}$), 1.65 m (1H, $CH_{2\text{cyclopropane}}$), 4.0 q (2H, CH_2), 1.0 t (3H, CH_3). Found, %: C 64.28, H 5.32, Cl 15.75. $C_{12}H_{13}O_2Cl$. Calculated, %: C 64.14, H 5.79, Cl 15.81.

2-p-Nitrophenyl-1-ethoxycarbonylcyclopropane (3). Yield 65%, bp 140–142°C (3 mm Hg), n_D^{20} 1.5610, d_4^{20} 1.30. ^{1}H NMR spectrum, δ, ppm: 7.50 m ($4H_{\text{arom}}$), 2.09 m (1H, $CH_{\text{cyclopropane}}$), 2.68 m (1H, $CH_{\text{cyclopropane}}$), 1.67 m (1H, $CH_{2\text{cyclopropane}}$), 1.63 m (1H, $CH_{2\text{cyclopropane}}$), 4.13 q (2H, CH_2), 1.12 t (3H, CH_3). Found, %: C 61.11, H 5.63, N 5.80. $C_{12}H_{13}O_4N$. Calculated, %: C 61.28, H 5.53, N 5.95.

Synthesis of 2-p-phenyl-substituted 1-hydroxymethyl-cyclopropanes. A 500-mL three-necked flask equipped with a power-driven stirrer, a dropping funnel, and a reflux condenser was charged with 9.1 g (0.25 mol) of $LiAlH_4$ in 200 mL of absolute diethyl ether, and 85 g (0.25 mol) of ethyl 2-phenylcyclopropanecarboxylate was added over a period of 2.0–2.5 h. The temperature of the reaction medium was maintained in the range 25–35°C. After the whole amount of the ester was added, the mixture was stirred for an additional 30 min. After that, distilled water and then 5% HCl were added dropwise. The ether layer was separated, and the aqueous layer was extracted with ether (2 × 20 mL). The ether extracts were combined with the ether layer and dried over calcined Na_2SO_4. After distillation of the diethyl ether, the products were isolated by vacuum distillation. Compounds 5 and 6 were prepared similarly.

The IR spectra of **4–6** contain absorption bands in the ranges 3095–3100 ($\nu_{\text{C–H ring}}$); 2980, 2940, 2880 ($\nu_{\text{C–H aliph}}$); 2900–3100 cm^{-1} (OH).

*2-Phenyl-1-hydroxymethylcyclopropane (**4**).* Yield 92%, bp 125–130°C (9 mm Hg), n_D^{20} 1.5110, d_4^{20} 1.335, MR_D = 43.235. ^{1}H NMR spectrum, δ, ppm: 6.92 m ($5H_{\text{arom}}$), 1.93 m (1H, $CH_{\text{cyclopropane}}$), 2.61 m (1H, $CH_{\text{cyclopropane}}$), 1.63 m (1H, $CH_{2\text{cyclopropane}}$), 1.65 m (1H, $CH_{2\text{cyclopropane}}$), 3.86 d (2H, CH_2O), 3.64 s (1H, OH). Found, %: C 81.25, H 8.05. $C_{10}H_{12}O$. Calculated, %: C 81.08, H 8.10.

*2-p-Chlorophenyl-1-hydroxymethylcyclopropane (**5**).* Yield 94%, bp 135–142°C (9 mm Hg), n_D^{20} 1.5225, d_4^{20} 1.478, MR_D = 48.998. ^{1}H NMR spectrum, δ, ppm: 7.14 m ($4H_{\text{arom}}$), 2.05 m (1H, $CH_{\text{cyclopropane}}$), 2.57 m (1H, $CH_{\text{cyclopropane}}$), 1.66 m (1H, $CH_{2\text{cyclopropane}}$), 1.72 m (1H, $CH_{2\text{cyclopropane}}$), 3.91 d (2H, CH_2O), 3.68 s (1H, OH). Found, %: C 65.20, H 6.75, Cl 19.94. $C_{10}H_{11}OCl$. Calculated, %: C 65.75, H 6.03, Cl 19.45.

*2-p-Nitrophenyl-1-hydroxymethylcyclopropane (**6**).* Yield 93%, bp 131–149°C (2 mm Hg), n_D^{20} 1.5190, d_4^{20} 1.524, MR_D= 49.998. ^{1}H NMR spectrum, δ, ppm: 7.55 m ($4H_{\text{arom}}$), 2.17 m (1H, $CH_{\text{cyclopropane}}$), 2.60 m (1H, $CH_{\text{cyclopropane}}$), 1.65 m (1H, $CH_{2\text{cyclopropane}}$), 1.69 m (1H, $CH_{2\text{cyclopropane}}$), 3.98 d (2H, CH_2O), 3.71 s (1H, OH). Found, %: C 62.22, H 5.40, N 7.80. $C_{10}H_{11}O_3N$. Calculated, %: C 62.18, H 5.69, N 7.25.

Synthesis of 2-p-phenyl-substituted cyclopropylmethyl methacrylates. A flask equipped with a reflux condenser was charged with 0.5 mol (74 g) of 2-phenyl-1-hydroxymethyl cyclopropane and heated in pyridine at 40°C for 2 h, after which 0.6 mol of methacryloyl chloride in 30 mL of absolute benzene was added dropwise. After the reaction completion, the liquid phase was separated and dried, the light fractions were distilled off, and the main fraction was distilled in a vacuum. Compounds 8 and 9 were prepared similarly.

The IR spectra of 7–9 contain absorption bands at 1435–1440, 3095–3100 ($\nu_{\mathrm{C–H\ ring}}$), 2880, 2940, 2980 ($\nu_{\mathrm{C–H\ aliph}}$), 750–760 ($\nu_{\mathrm{C–Cl}}$), 1365 and 1335 cm^{-1} (NO_2).

2-Phenylcyclopropylmethyl methacrylate (7). Yield 95%, bp 95–110°C (3 mm Hg), n_D^{20} 1.4040, d_4^{20} 0.878, $MR_D = 60.027$. ^{1}H NMR spectrum, δ, ppm: 7.25 m ($5H_{\mathrm{arom}}$), 1.91 m (1H, $CH_{\mathrm{cyclopropane}}$), 2.58 m (1H, $CH_{\mathrm{cyclopropane}}$), 1.58 m (1H, $CH_{2\mathrm{cyclopropane}}$), 1.60 m (1H, $CH_{2\mathrm{cyclopropane}}$), 4.08 d (2H, CH_2O), 5.48 s (1H, $CH_{2\mathrm{vinyl}}$), 5.89 s (1H, $CH_{2\mathrm{vinyl}}$), 1.57 s (3H, CH_3–C=). Found, %: C 77.45, H 7.83. $C_{14}H_{16}O_2$. Calculated, %: C 77.78, H 7.40.

*2-p-Chlorophenylcyclopropylmethyl methacrylate (**8**).* Yield 96%, bp 110–115°C (3 mm Hg), n_D^{20} 1.4105, d_4^{20} 0.970, $MR_D = 63.787$. ^{1}H NMR spectrum, δ, ppm: 7.36 m ($4H_{\mathrm{arom}}$), 1.88 m (1H, $CH_{\mathrm{cyclopropane}}$), 2.63 m (1H, $CH_{\mathrm{cyclopropane}}$), 1.58 m (1H, $CH_{2\mathrm{cyclopropane}}$), 1.64 m (1H, $CH_{2\mathrm{cyclopropane}}$), 4.11 d (2H, CH_2O), 5.49 s (1H, $CH_{2\mathrm{vinyl}}$), 5.92 s (1H, $CH_{2\mathrm{vinyl}}$), 1.66 s (3H, CH_3–C=). Found, %: C 67.24, H 5.57, Cl 14.81. $C_{14}H_{15}O_2Cl$. Calculated, %: C 67.07, H 5.98, Cl 14.17.

*2-p-Nitrophenylcyclopropylmethyl methacrylate (**9**).* Yield 94%, bp 117–125°C (3 mm Hg), n_D^{20} 1.4080, d_4^{20} 0.950, $MR_D = 67.585$. ^{1}H NMR spectrum, δ, ppm: 7.60 m ($4H_{\mathrm{arom}}$), 2.18 m (1H, $CH_{\mathrm{cyclopropane}}$), 2.70 m (1H, $CH_{\mathrm{cyclopropane}}$), 1.66 m (1H, $CH_{2\mathrm{cyclopropane}}$), 1.72 m (1H, $CH_{2\mathrm{cyclopropane}}$), 4.22 d (2H, CH_2O), 5.66 s (1H, $CH_{2\mathrm{vinyl}}$), 6.01 s (1H, $CH_{2\mathrm{vinyl}}$), 1.89 s (3H, CH_3–C=). Found, %: C 64.73, H 5.18, N 5.66. $C_{14}H_{15}O_4N$. Calculated, %: C 64.36, H 5.75, N 5.36.

To cross-link the copolymers, we prepared 2–10% polymer solutions and applied them onto a 60 × 90 mm glass support by centrifugation at 2500 rpm. After that, the photoresist was kept for no less than 20 min to enhance its adhesion to the support. Then, the parts of the photoresist film protruding beyond the support were cut off, avoiding the film peel-off. The photoresist thickness was measured with an MII-4 Linnik microinterferometer. The resist film thickness after drying for 10 min at room temperature and then for 20 min at 25°C/10 mmHg was 0.2–0.3 μm.

The pieces with the deposited photoresist were exposed on an installation with a point light source through a phototemplate.

The development was performed in a jet installation. A 1 : 2 mixture of dioxane with isopropyl alcohol at 18–25°C was used as a developer. The extent of the photochemical polymerization (cross-linking) of photoresist base molecules under UV irradiation served as a criterion of the photosensitivity of the negative photoresists.

A DRT-220 mercury lamp was used as a UV radiation source (current 2.2 A, distance from the radiation source 15 cm, velocity of the exposure meter shutter 720 mm h^{-1}, exposure time 5–20 s). The content of the insoluble polymer was calculated from the residue weight.

The photosensitivity is a quantity reciprocal to the UV dose absorbed by the photoresist, i.e., to the dose required for converting the photoresist to the insoluble state. It is measured in cm^2 W^{-1} s^{-1} = cm^2 J^{-1}:

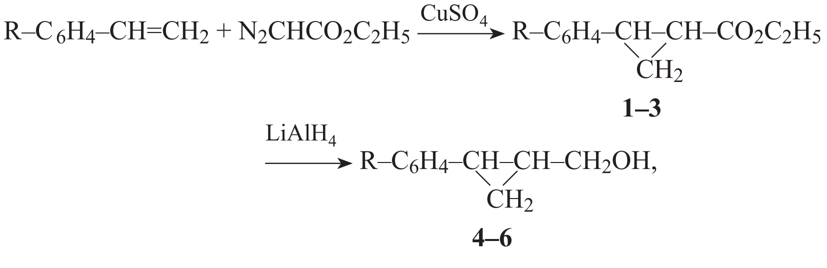
$$S = \frac{1}{H} = \frac{1}{E\tau},$$

where H is the exposure (or UV irradiation dose) (J cm); E, intensity (W cm^{-2}); and τ, irradiation time (s).

RESULTS AND DISCUSSION

First, we studied the compounds formed by the reactions of ethoxycarbonylcarbene with styrene and *p*-substituted styrenes under the conditions of thermocatalytic decomposition of ethyl diazoacetate in the presence of anhydrous $CuSO_4$ (Scheme 1).

Scheme 1.



$$\mathrm{R{-}C_6H_4{-}CH{=}CH_2 + N_2CHCO_2C_2H_5 \xrightarrow{CuSO_4} R{-}C_6H_4{-}\underset{CH_2}{CH{-}CH}{-}CO_2C_2H_5}$$

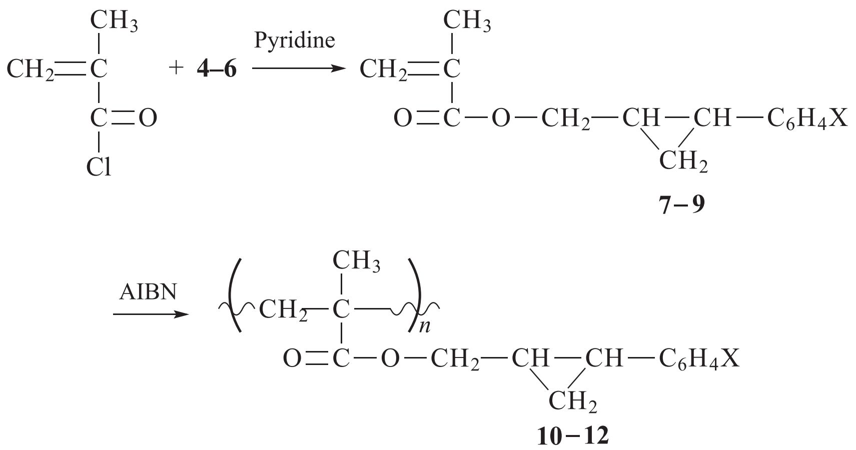
1–3

$$\mathrm{\xrightarrow{LiAlH_4} R{-}C_6H_4{-}\underset{CH_2}{CH{-}CH}{-}CH_2OH,}$$

4–6

R = H (**1**, **4**), Cl (**2**, **5**), NO_2 (**3**, **6**).

Scheme 2.



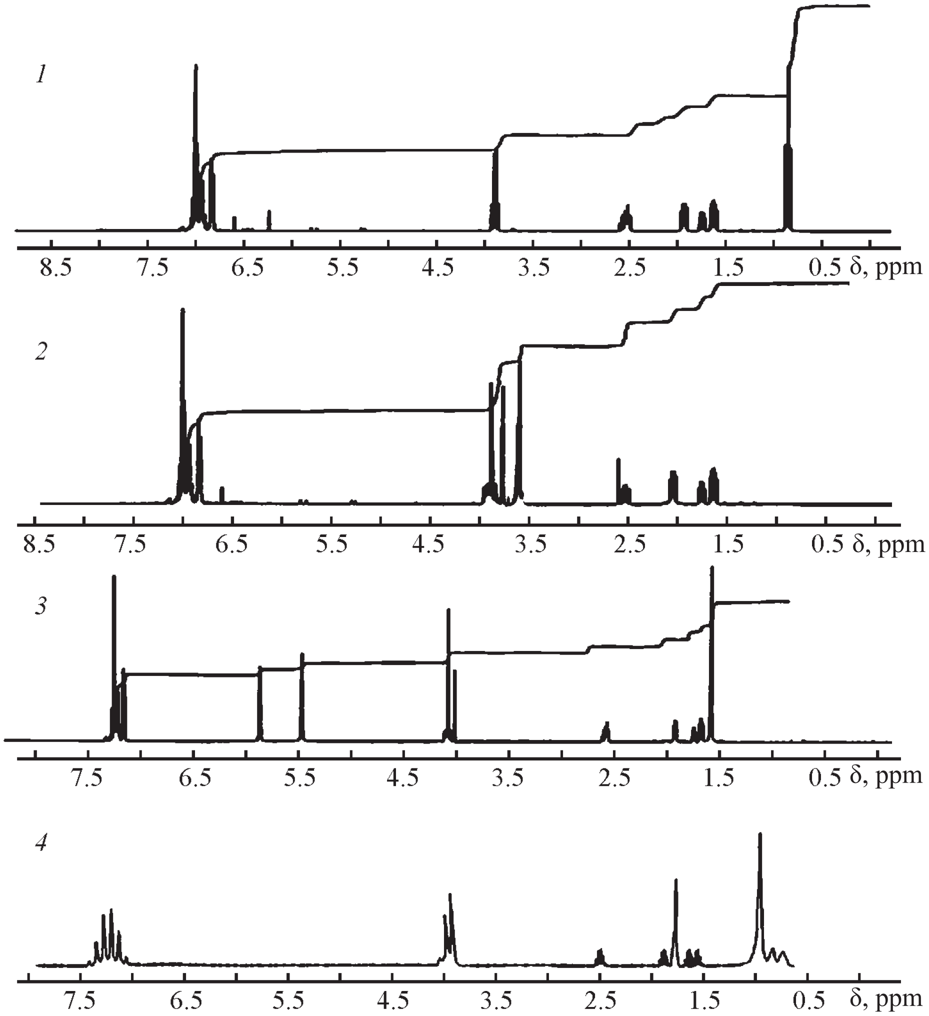


Fig. 1. NMR spectra of the synthesized compounds: (*1*) 2-phenyl-1-ethoxycarbonylcyclopropane, (*2*) 2-phenyl-1-hydroxymethylcyclopropane, (*3*) 2-phenylcyclopropylmethyl methacrylate, and (*4*) poly(2-phenylcyclopropylmethyl methacrylate).

The IR spectra and GLC data show that the compounds synthesized are mixtures of two geometric isomers, cis and trans (relative to the three-membered ring); the ratio of these isomers is *trans : cis* = 70 : 30. The product purity was checked by GLC and was higher than 99.2% in all the cases.

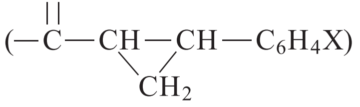
Phenyl and *p*-substituted phenyl derivatives of cyclopropylmethyl methacrylate (**7–9**) were prepared by the reaction in Scheme 2.

The radical polymerization was performed in the bulk and in a benzene solution in sealed ampules at 70°C in the presence of azobis(isobutyronitrile) (AIBN) (0.3% relative to the monomer weight). The polymers obtained are white powders soluble in aromatic and chlorinated hydrocarbons and insoluble in aliphatic hydrocarbons and lower alcohols.

The structure of the polymers obtained was determined by IR and NMR spectroscopy and by elemental analysis. The IR spectra of the polymers contain the carbonyl absorption bands at 1730 cm^{-1}, compared to 1720 cm^{-1} in the spectra of the initial monomers. The shift of the C=O absorption maximum is due to opening of the double bond with the break of the conjugation and electron density redistribution in the system.

In the spectra of the polymers obtained from monomers **7–9**, the stretching and bending vibrations of the benzene rings are observed at 1608, 1580, 1496, and 1456 cm^{-1}. The absorption bands at 1035–1040 cm^{-1}, observed in the IR spectra of both the monomers and polymers, can be assigned to cyclopropane groups. The bands in the regions of 900–1000 and 1640–1645 cm^{-1}, belonging to bending and stretching vibrations of the vinyl double bond and observed in the spectra of the monomers, disappear in the spectra of the polymers. In the NMR spectra of the polymers (Fig. 1), there are well-defined proton signals of the benzene (δ = 6.80–7.60 ppm) and cyclopropane (δ = 0.25–2.60 ppm) rings; the vinyl proton signals (δ = 4.50–5.05 ppm) are absent in the spectra of the polymers.

Thus, the spectroscopic analysis shows that the polymerization occurs exclusively via the double bond of the vinyl group, with the pendant reactive fragments



$(-\overset{O}{\overset{\|}{C}}-\underbrace{CH-CH}_{CH_2}-C_6H_4X)$ remaining unchanged.

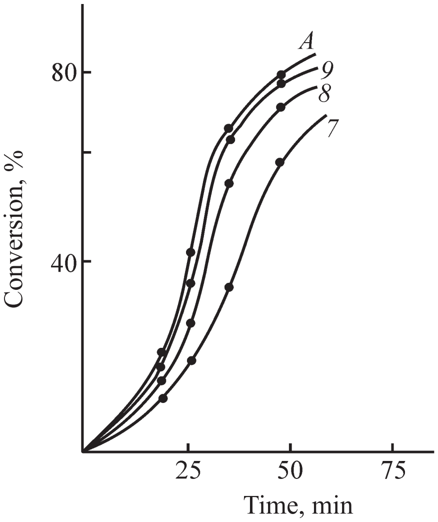


Fig. 2. Conversion as a function of the polymerization time. The curve nos. are compound nos.: (*7*) 2-phenylcyclopropylmethyl methacrylate, (*8*) 2-*p*-chlorophenylcyclopropylmethyl methacrylate, (*9*) 2-*p*-nitrophenylcyclopropylmethyl methacrylate, and (*A*) *p*-chlorophenyl methacrylate.

To evaluate the polymerization ability of monomers **7–9**, we studied the dependence of the polymer yield on the polymerization time under the chosen standard conditions. Figure 2 shows the kinetic curves of the polymerization of monomers **7–9** and *p*-chlorophenyl methacrylate (*A*). The polymerization of chlorophenyl methacrylate occurred in the bulk at 70°C under similar conditions. Comparison of the kinetic curves shows that the synthesized monomers **7–9** and *p*-chlorophenyl methacrylate appreciably differ in the polymerization

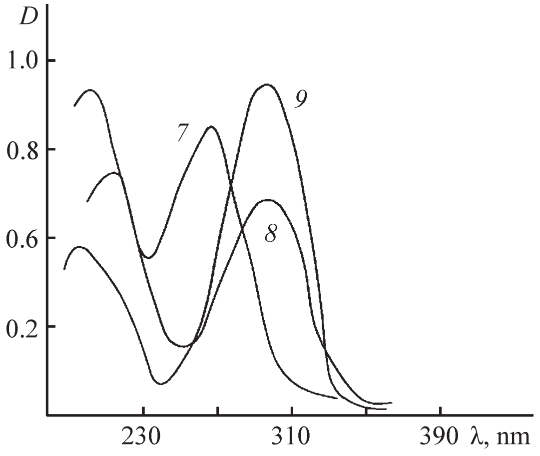
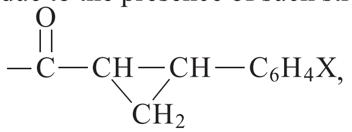
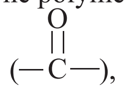
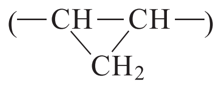


Fig. 3. UV spectra of the synthesized compounds. The curve nos. are compound nos.: (*7*) 2-phenylcyclopropylmethyl methacrylate, (*8*) 2-*p*-chlorophenylcyclopropylmethyl methacrylate, and (*9*) 2-*p*-nitrophenylcyclopropylmethyl methacrylate.

ability and can be ranked in the following order: *A* > **9** > **8** > **7**. This fact is probably associated with the effect of the chlorophenyl group via O atom on the carbonyl group conjugated with the vinyl moiety.

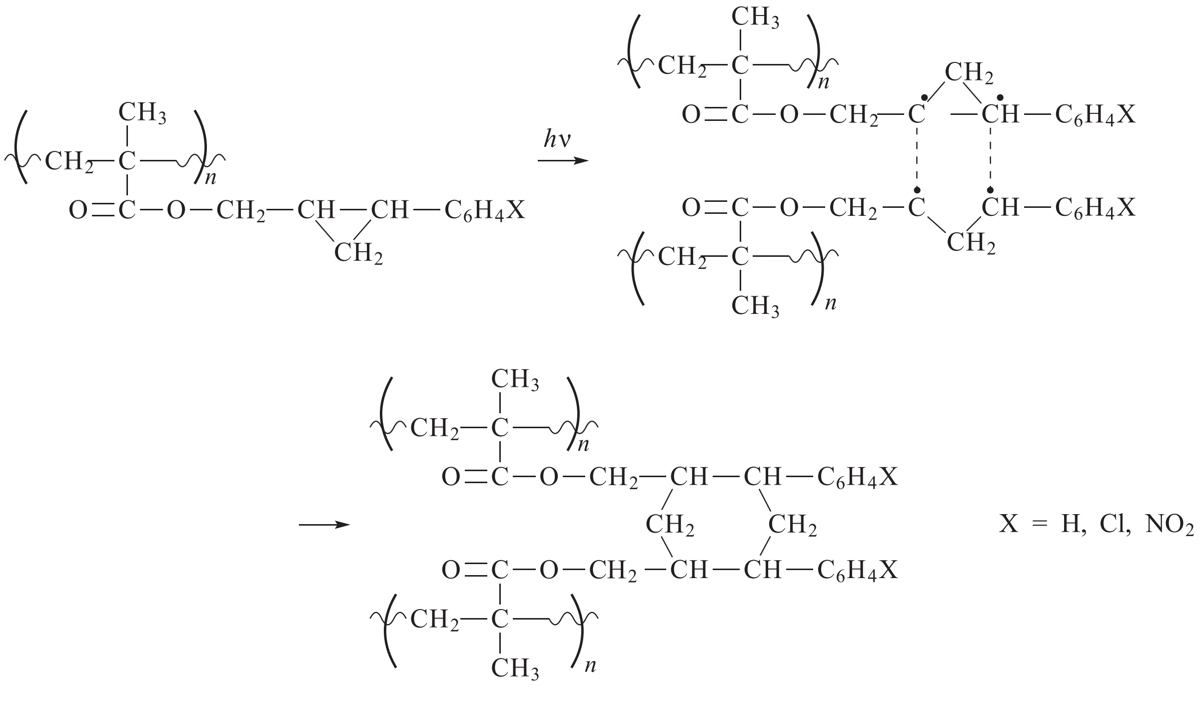
The observed order of the monomer activity in radical polymerization is not directly associated with the conjugation effect, although the UV spectra (Fig. 3) of these monomers demonstrate changes in the absorption band. Probably, steric factors play an important role in the chain propagation step. The relative stability of the radical responsible for the chain propagation can be another important factor influencing the course of the polymerization.

As we found, the radical polymerization of all the monomers in the bulk is accompanied by self-acceleration. The onset of the self-acceleration is observed at ~25% conversion of the monomers, in agreement with the data of [10].

The synthesized polymers containing UV-sensitive reactive groups are valuable objects for photochemical studies and can serve as a basis for the development of photosensitive materials. The presence of reactive groups in the polymer macromolecules makes it interesting to study the photochemical cross-linking of the polymer under the action of UV radiation. Intense occurrence of photochemical processes in the polymer is due to the presence of such strongly absorbing groups as , which enhance the UV sensitivity of the polymer and are prone to photochemical transformations leading to the polymer chain cross-linking. The carbonyl group is the main functional group in the polymer. The polymer contains various absorbing groups such as  $(-\overset{O}{\overset{||}{C}}-)$, Cl, and . Therefore, such polymers should be photosensitive. The UV irradiation of the polymer films leads to opening of the cyclopropane rings and their recombination with the probable formation of the cyclohexane ring acting as a cross-linking group. In the process, the polymer becomes insoluble. Presumably, the reaction occurs by the mechanism described in [1, 11].

The cross-linking of macromolecular chains under the action of actinic radiation occurs considerably faster than the oxidation. We performed experiments with different polymer concentrations in thin films. The UV spectra of the polymer films contain absorption bands at ~275 and ~285 nm. To determine the pathways and mechanism of the photochemical reaction, we

Scheme 3.



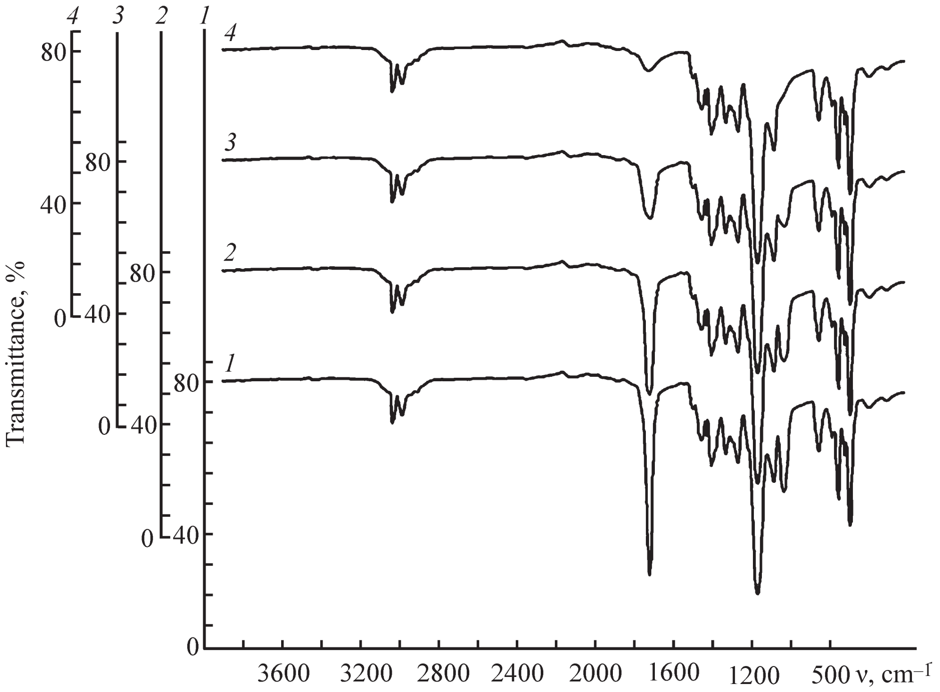


Fig. 4. IR spectra of the poly(2-phenylcyclopropylmethyl methacrylate) film, (*1*) unirradiated and irradiated for (*2*) 5, (*3*) 10, and (*4*) 25 s.

studied the IR spectra (Fig. 4) in various steps of the UV irradiation. The UV irradiation leads to a decrease in the intensity and then to the disappearance of the absorption bands at 1720 and 1035 cm^{-1}, corresponding to the carbonyl and cyclopropane groups in the pendant chains of the macromolecule. This fact suggests that the photochemical cross-linking occurs via opening of the cyclopropane ring with the participation of the carbonyl group. Our studies demonstrate the possibility of the synthesis of a new valuable photosensitive polymer (35–40 cm^2 J^{-1}) as a base of a photosensitive material. The film thickness is an important factor in irradiation. Good results were obtained in experiments with 0.2–0.3 μm thick films. After irradiation for 30–80 s (40–75% conversion), the polymer films become insoluble in organic solvents in which they dissolved at room temperature before irradiation.

The synthesized polymers also have good optical characteristics (n_D^{20} 1.5130–1.5215) and remain transparent on heating up to 100°C for 1–1.5 h. The light transmittance in the visible range is 85–90% and depends only slightly on the sample thickness up to 6–8 mm; at higher thickness, the optical properties become somewhat worse, which is typical of all the known transparent materials.

The synthesized polymers can be used as photosensitive polymer materials, and also in optics as optically transparent materials.

CONCLUSIONS

(1) New polyfunctional monomers, cyclopropyl methacrylates, were synthesized, their radical homopolymerization was performed, and the composition and structure of the polymers based on the corresponding monomers was determined.

(2) UV-induced cross-linking of the polymers was studied. The synthesized polymers exhibit relatively high photosensitivity (35–40 cm^2 J^{-1}) and are good optically transparent materials.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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