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Synthesis and radical copolymerization of 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate with maleic anhydride

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Abstract

A monomer containing a photochromic group was synthesized by condensation of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene with cinnamoyl chloride. The structure and composition of the monomer were established by FTIR, UV–Vis, ^{1}H NMR spectroscopy, and elemental analysis. The values of chemical shifts, multiplicity, and integrated intensity of 1H signals in one-dimensional NMR spectra of the monomer were determined, and the homo- and heteronuclear interactions were found. The crystal structure of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene was established using X-ray diffraction analysis. A copolymer containing photochromic mesogenic fragment was synthesized by radical copolymerization of 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate with maleic anhydride.

KEYWORDS
4-(2-hydroxyethyloxy)-4-cyanoazobenzene, azobenzene, copolymer, cyanoazobenzene, monomer

1 | INTRODUCTION

Over the past few years, liquid crystal (LC) polymers, containing mesogenic and photochromic groups, have attracted the attention of researchers as one of the most promising light-controlled “smart materials.”[1,2]

Previously, the preparation of photochromic comb-shaped LC polymers with photochromic azobenzene groups was carried out by various authors.[3-6] As photoactive fragments, the derivatives of azobenzene, cinnamates, and coumarin were used.[7,8]

Among the existing three main structural modifications of LC polymers—nematic, smectic, and cholesteric types—of particular interest are cholesteric LC polymers with a spiral supramolecular structure and unique optical properties: selective light reflection, high optical activity, etc. These optical properties determine the area of their practical use as selective filters, polarizing light reflectors, color photo-controlled films and coatings, systems for recording information, etc..[9-11]

A wide range of applications of LC polymers is predicted in.[12] In particular, the stored holographic and digital information is due to the transformation of mesophases. The first report of Wendorf and Eich[13] was devoted to the holographic effects of accumulation with an azobenzene-containing compound. It was shown[14] for the first time that the reconstructed image as obtained from coherent illumination of the hologram is reversibly stored in the LCP cell. The hologram remained unchanged after more than 2 years of storage at room temperature.

Introduction of photochromic fragments, such as azobenzene dye molecules undergoing cis-trans isomerization under the influence of light, provides their photo-optical sensitivity and opens up the possibility of obtaining a new photosensitive and photochromic polymers. The photosensitive compounds undergoing photoisomerization contain double bonds, for example, —C=C-stylbenes and helices, azomethines, olefins, salicylidenanilines, and others.[15] Ruhmann et al[16] described the synthesis of LC copolymethacrylates with an azobenzene moiety. The ability of azo groups undergoing photochemical trans-cis isomerization was shown. The cis-trans isomerization of azo groups is used in preparation of light-sensitive materials for recording and storing information.[16,17]

In the present communication, we report the synthesis and study of cyanoazobenzene compounds 2-(4-((4-isocyanophenyl)diazenyl) phenoxy)ethyl-3-phenylacrylate, and its copolymer with a maleic anhydride, containing the photochromic groups, which can be considered as the promising light-sensitive materials, was first synthesized.

2 | MATERIALS AND METHODS

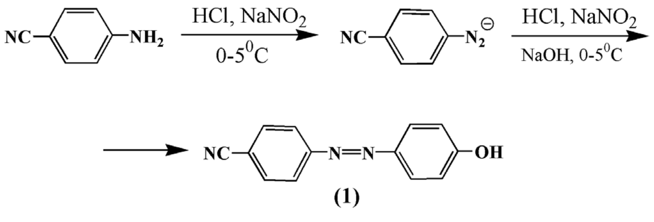
2.1 | Materials

4-Aminobenzonitrile (purity 98%) and cinnamoyl chloride (purity98%) were purchased from Sigma Aldrich (Saint Luis, MO, USA) and used as received. Sodium nitrite ($NaNO_2$, purity99.7%, AppliChem GmbH), sodium hydroxide (NaOH, purity97%), potassium carbonate (K_2CO_3, purity99%), potassium iodide (KI), and phenol were purchased from LaborFarm (Almaty, Kazakhstan). Maleic anhydride (purity99%, Fluka) was purchased from Labtech (Moscow, Russia). All organic solvents used in this study were purified by distillation.

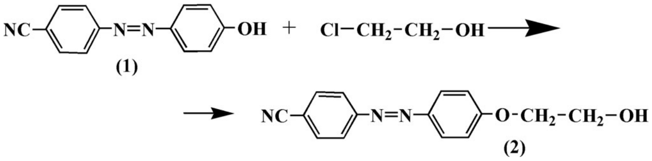
2.2 | Synthesis of compounds containing photochromic groups

Synthesis of 4-hydroxy-4-cyanoazobenzene (1) was provided by Scheme 1 according to the procedure described in Reference 17. Aqueous solution, 30 ml, of $NaNO_2$ (0.045 mol) was dropwise added to 14 ml solution of 4-aminobenzonitrile (0.045 mol) and concentrated HCl at 5°C. Then, at the same temperature, a solution of phenol (0.050 mol) in aqueous NaOH (2 M, 75 ml) was added. The mixture was mixed for 2 h. The resulting sediment was filtered and washed by water up to a neutral state. The filtered product was recrystallized from ethanol and dried in a vacuum oven.

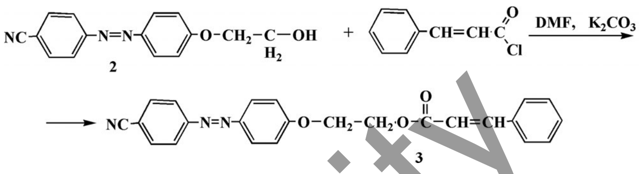
Scheme 2 shows the synthesis of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) according to the procedure described in Reference 18. 4-hydroxy-4-cyanoazobenzene, 4.8 g (0.02 mol), 1.62 g (0.02 mol) of ethylene hydrochloride, 3.32 g (0.024 mol) of potassium carbonate, and 0.332 mol KI (0.002 mol)were dissolved in 140 ml of DMF in a two-throated flask. After complete dissolution of the solids in DMF, the reaction mixture was boiled and intensively stirred for 24 h. The temperature was kept at 90°C. In the final stage, the hot reaction mixture was poured into 500 ml of ice water. A cherry-red precipitate was formed. The precipitate was filtered, recrystallized from 2-propanol, and dried in a vacuum oven up to constant weight. The obtained target product is an orange-colored powder with a melting temperature of 160°C.



SCHEME 1 Synthesis of 4-hydroxy-4-cyanoazobenzene (1)



SCHEME 2 Synthesis of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2)



SCHEME 3 Synthesis of 2-(4-((4-isocyanophenyl)diazenyl) phenoxy)ethyl-3-phenylacrylate (3)

2.3 | Synthesis of monomer

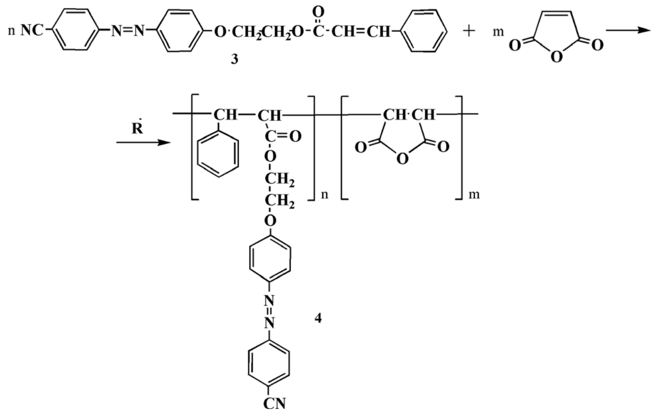
Synthesis of 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate (3) was performed by reacting 4-(2-hydroxyethyloxy)-4-cyanoazoxybenzene (2) with cinnamoyl chloride in the presence of potassium carbonate in DMF (Scheme 3). The reaction mixture was stirred on a magnetic stirrer at a temperature of 60–70°C for 24 h. The salt precipitate was filtered out, the monomer was precipitated with water, an orange-colored product was filtered, washed several times with water, and dried at room temperature. The yield of the monomer product **3** is 70% with a melting temperature of 174°C.

2.4 | Synthesis of copolymer

A photochromic polymer **4** was synthesized according to Scheme 4. Monomer (3) and maleic anhydride were placed in an ampoule, dissolved in DMF, and an initiator AIBN (2%) was added. Then, the reaction mixture was purged with argon for 15 min, the ampoule was sealed, and placed in a thermostat heated to 80°C. After completion of the polymerization (after 48 h), the ampoule was cooled and opened. The resulting copolymer was precipitated in ethyl alcohol.

2.5 | Methods

FTIR spectra were registered on a FSM-1201 spectrophotometer (LLC Infraspek, Russia) in KBr pellets in the range of wave numbers from 500to 400 cm^{-1}.



SCHEME 4 Radical copolymerization of monomer (3) with maleic anhydride

The X-ray diffraction experiments were performed on Bruker D8 Venture Kappa Duo PHOTON100 by IμS micro-focus sealed tube with MoKα (0.71073 Å) radiation at a temperature of 120(2)K.

Electronic absorption spectra were recorded on a UV-1800 Shimadzu dual-beam scanning spectrophotometer (Shimadzu, Kyoto, Japan) in 10 mm thick quartz cuvettes. Spectra were obtained at 25 °C, with resolution of 1 nm.

Chromatography-mass spectra were recorded on Agilent 7890A instrument with a 5975C mass-selective detector inert MSD with Tropl-Axis Detector G1371A (Agilent Technologies, Inc., Wilmington, USA).

NMR spectra were recorded on a JNN-ECA Jeol 400 spectrometer using a DMSO-d_6 solvent (BrukerDaltonics, Japan).

Thermogravimetric(TG), differential thermal (DTG), and differential scanning calorimetric (DSC) analyses were carried out by using DTA/DSC equipment (LabsysEVO, Setaram, France) in dynamic mode in the temperature range of 30–500°C at a heating rate of 10^0C/min in nitrogen atmosphere and air.

3 | RESULTS AND DISCUSSION

The spatial structure of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) was first established by X-ray diffraction analysis. The spatial structure and packaging of molecule **2** are shown in Figures 1 and 2. The physical and chemical constants of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) coincide well with previously obtained data.[18]

X-ray diffraction analysis of compound 2 ($C_{15}H_{13}N_3O_2$) and all relevant crystallographic data are given in Table 1 and Figures 1 and 2. The structure was identified by direct methods (XT)[19] and refined by full-matrix least squares based on F^2 (SHELXL2018).[20] The hydrogen atoms on carbon were fixed into idealized positions (riding model), and hydrogen atoms on oxygen O1 and nitrogen N5 were found on difference Fourier map and refined under rigid body assumption with assigned temperature factors $H_{iso}(H) = 1.2\ U_{eq}$.X-ray crystallographic data have been deposited with the Cambridge Crystallographic Data Centre (2053997) and can be obtained free of charge from the center via its website (www.ccdc.cam.ac.uk/getstructures). X-ray diffraction analysis showed that the 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) molecule is flat and the molecules are connected by intermolecular hydrogen bonds between O1 and N5 atoms.

Synthesis of a monomer (**3**) was carried out by condensation of4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) with cinnamoyl chloride in anhydrous 50 ml of DMF at 90–100°C for 48 h as shown in Scheme 3. The progress of the reaction and the purity of the obtained compounds were monitored by thin-layer chromatography on Silufol UV-254 plates. The monomer was isolated by column chromatography on alumina. Photochromicazobenzene-containing monomer **3** orange crystalline substance with a yield of 51%, m.p. = 172-174°C. The structure of the monomer (**3**) was confirmed by elemental analysis, FTIR, ^1H NMR spectroscopy, and HPLC analysis.

$C_{24}H_{19}N_3O_3$ (397.43) Calc. C 72.46 H 4.78 N 10.56.

Found. C 72.65 H 4.93 N 10.89.

The mass spectrum of the monomer – 2-(4-((4-isocyanophenyl) diazenyl)phenoxy)ethyl-3-phenylacrylate (3) is presented in Figure 3.

The retention time for the characteristic components of monomer 3 is 14.26 min (4-aminobenzonitrile) and 14.011 min (trans-Cinnamic acid) with a probability of 90% and 96%, respectively.

The structure of compound 3 was confirmed by two-dimensional NMR spectra (Figures 4-6). Spin–spin interactions were detected using the COSY spectrum. Heteronuclear correlations of HC through a single bond are established by means of the HMQC spectrum (^1H-^{13}C).

From an analysis of ^1H NMR spectra of the monomer 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate (3), it is possible to assume the presence in the solution of several N—CO and CO—CH═CH— rotational bonds C_6H_5 isomers. Since the barriers of these rotations are small, they can lead both to the registration of spectra from several conformers and simply to a substantial broadening of the lines of the spectrum. In some cases, this did not allow the unambiguous assignment of signals.

The ^1H NMR spectrum of monomer 3 shows two-proton doublet signals at 3.72 and 4.07 ppm with the same ^3J 4.4 Hz of ethylene

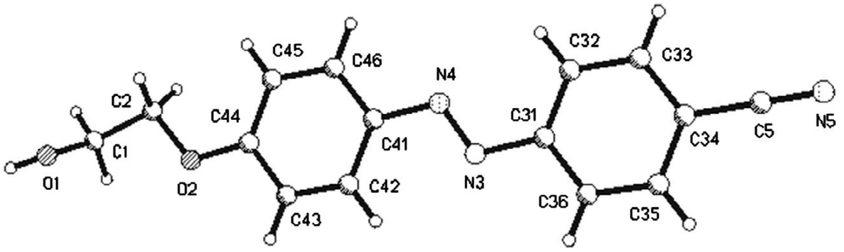


FIGURE 1 Spatial structure of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2)

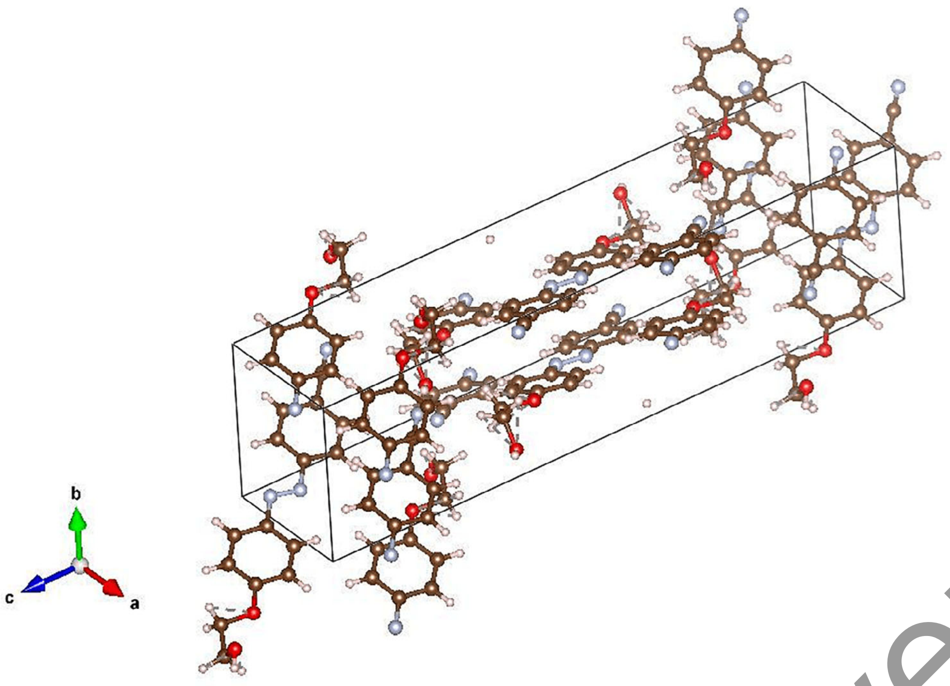


FIGURE 2 Packing molecule 4-(2-hydroxyethyloxy)-4-cyanoazobenzene (2) in the crystal

TABLE 1 Experimental data for single-crystal X-ray diffraction analysis of 4-(2-hydroxyethyloxy)-4-cyanobenzene (2)

Parameter	2
Formula	$C_{15}H_{13}N_3O_2$
Formula mass	267.28
Crystal system	Monoclinic
Space group	$P2_1/c$ (No. 14)
a/Å	5.9195(2)
b/Å	7.5642(3)
c/Å	28.0287(11)
α/°	90
β/°	92.340(1)
γ/°	90
V/Å^3	1253.97(8)
Z	4
$D_{(calc)}$/g ml $^{-1}$	1.416
$\mu_{(MoKa)}$ /mm^{-1}	0.097
$F_{(000)}$	560
Crystal size/mm	0.04 × 0.19 × 0.48
Temperature/K	120(2)
Radiation/Å	0.71073
θ/° (min-max)	2.8–27.5
Index ranges	−7.7; −9.8; −36.36
(measured/unique/observed)	17,269/2890/2336
Data/restrains/parameters	2890/0/188
Goodness-of-fit on F^2	1.063
Final R, wR2	0.0410, 0.1106
R indices (all data)	0.0536, 0.1171
Largest diff. peak and hole	0.33, −0.25

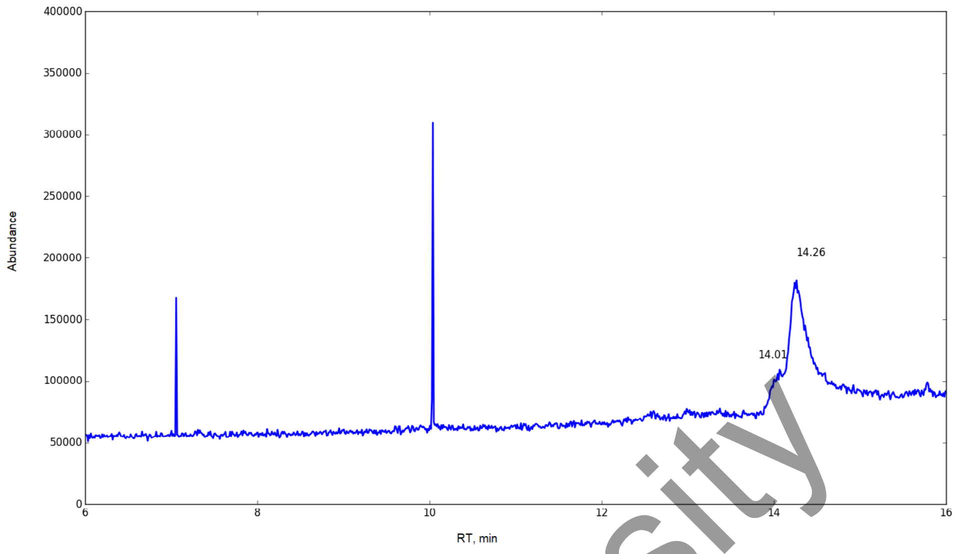
protons $H^{9,9}$ and $H^{8,8}$, respectively. Due to the presence of protons with close chemical shifts, the remaining protons appeared as multiplets at 6.5–6.8 (1H, H^{21}), 7.09–7.12 (4H, $H^{2,6,25,27}$), 7.32–7.67 (4H, $H^{22,24,26,28}$), 7.84–7.92 (5H, $H^{3,5,17,19,22}$), and 7.97–7.99 (2H, $H^{16,20}$) ppm.

In the ^{13}C NMR spectrum of monomer 3, the signals of ethylene carbon atoms appear at 59.95 (C^9) and 70.69 (C^8) ppm. The carbon atoms of the cyanophenyl fragment resonate at 118.15 (C^{18}), 119.04 (C^{29}), 128.26 ($C^{16,20}$), 134.25 ($C^{17,19}$), and 154.60 (C^{15}) ppm. The aromatic carbon atoms of the diazenylphenoxy group appear at 163.00 (C^1), 115.75 ($C^{2,6}$), 125.82 ($C^{3,5}$), and 145.59 (C^4) ppm. The carbon atoms of the phenyl acrylate fragment resonate at 115.86 (C^{21}), 128.98 ($C^{24,28}$), 129.31 ($C^{25,27}$), 135.64 (C^{23}), 146.56 (C^{22}), and 166.71 (C^{11}) ppm.

The structure of compound **3** was also confirmed by two-dimensional NMR spectroscopy of COSY (^1H-^1H) and HMQC (^1H-^{13}C), which allow to establish spin–spin interactions of homo- and heteronuclear nature. The observed correlations in the molecule are shown in Figure 4. In the spectra of ^1H-^1H COSY compounds, spin–spin correlations are observed through three proton bonds of neighboring methylene–methylene and methine–methine groups H^8–H^9 (3.71, 4.06 and 4.06, 3.71), H^{21}–H^{22} (6.52, 7.37 and 7.37, 6.52; 6.62, 7.63 and 7.63, 6.62), $H^{25,27}$–$H^{26,28}$ (7.09, 7.87, and 7.87, 7.09), and $H^{17,19}$–$H^{16,20}$ (7.88, 7.95 and 7.95, 7.88) ppm (Figure 5). Heteronuclear interactions of protons with carbon atoms via a single bond were established using ^1H-^{13}C HMQC spectroscopy for the following pairs present in the compound: H^9–C^9 (3.71, 60.08), H^8–C^8 (4.06, 70.71), $H^{2,6}$–$C^{2,6}$ (7.12, 115.89), $H^{24,28}$–$C^{24,28}$ (7.36, 129.18) and $H^{17,19}$–$C^{17,19}$ (7.96, 134.17) ppm (Figure 6).

Thus, the combination of FTIR, ^1H, ^{13}C NMR spectroscopy, and chromatographic analysis confirms the structure of monomer 3 that further was involved in copolymerization reaction with maleic anhydride to obtain copolymer 4.

FIGURE 3 Chromatogram of the monomer 2-(4-((4-isocyanophenyl) diazenyl)phenoxy)ethyl-3-phenylacrylate (3)



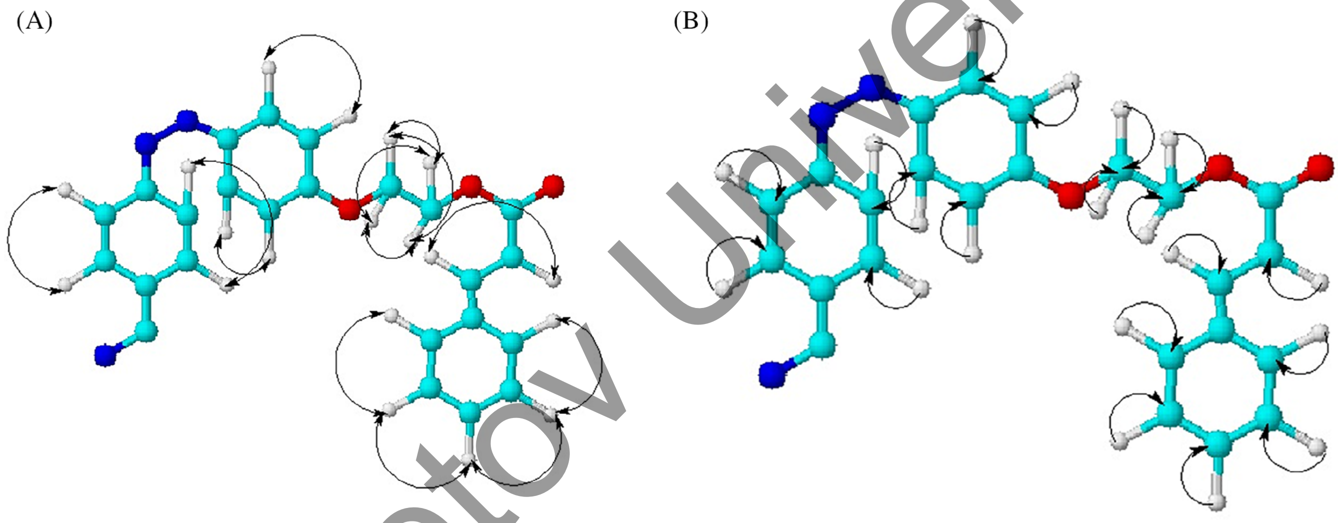


FIGURE 4 a) correlation (^1H-^1H) COSY, b) correlation (^1H-^{13}C) HMQC monomer - 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate (3)

UV spectra of maleic anhydride, monomer 3, and copolymer in ethanol are compared in Figure 7.

The UV spectrum of monomer **3** is characterized mainly by a low-intensive band in the region of 255–260 nm. The band at λ_{max} 260 nm belongs to the symmetric π–π^* junction of the aromatic ring and is called KIV bands, which are caused by transitions in the benzene ring. An increase in the absorption intensity in the K-band region may be associated with an increase in the excited molecules. The strong absorption band at λ_{max}360–365 nm corresponds to the π–π^* transition and the n–π^* transition in azochromophore.[21] Copolymer 2 also exhibits the absorption band at λ_{max} 360–365 nm. An increase in absorption at 352–460 nm indicates photoisomerization from *trans* to *cis* form. Consequently, light irradiation in this wavelength region should cause photoisomerization from *trans* form to *cis* form and vice versa.[22] The presence of a double C═C bond renders the compounds photochromic properties, when exposed to UV light, manifesting *E/Z* isomerization. As seen from Figure 6, the wide and intense absorption band in the region of 200–300 nm corresponds to the absorption band of maleic anhydride. In the UV spectrum of copolymer 2, this band disappears and confirms the involvement of maleic anhydride in copolymer 2 composition. The strong absorption band at λ_{max} 360 nm is characteristic of azochromophoric groups of copolymer 2.

It has been reported that azobenzene molecule aligns perpendicular to the plane of linearly polarized light. Such photo-induced orientation allows to write, erase, and rewrite optical information on the side chain of LC or on amorphous polymer films containing azobenzene group.[15]

According to TG and DTG data, decomposition of copolymer 4 takes place in the temperature range of 300 –370°C and is accompanied by weight loss (Figure 8). Full destruction of the sample is completed at 590°C with total weight loss of 92.23%.

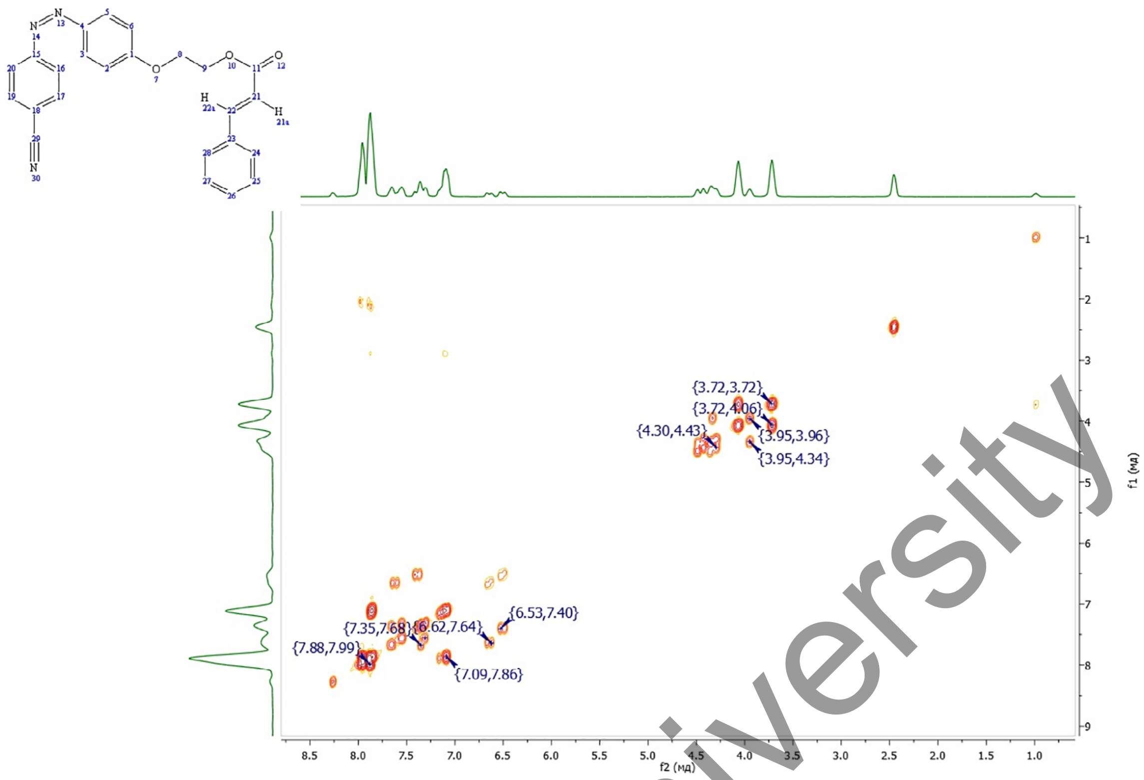


FIGURE 5 NMR (^{1}H-^{1}H) COSY monomer spectrum - 2-(4-((4-isocyanatophenyl)biphenyl)phenoxy)ethyl-3-phenylacrylate (3)

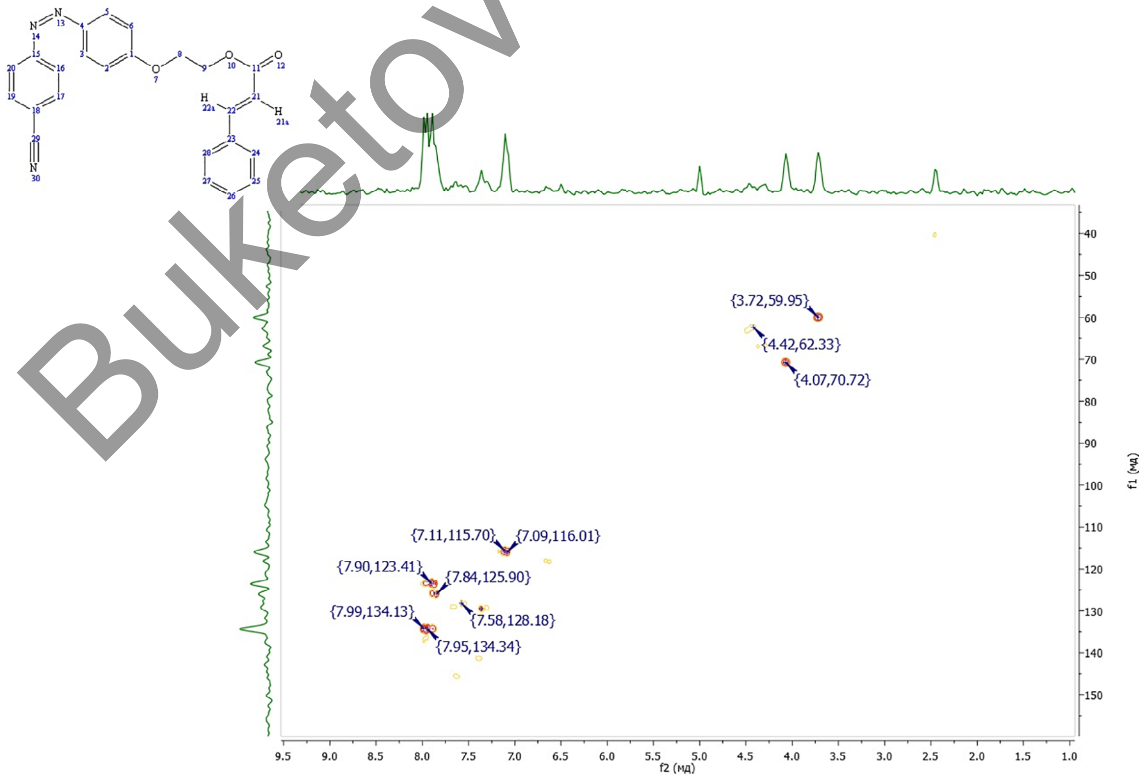


FIGURE 6 NMR (^{1}H-^{13}C) HMQC monomer spectrum - 2-(4-((4-isocyanatophenyl)biphenyl)phenoxy)ethyl-3-phenylacrylate (3)

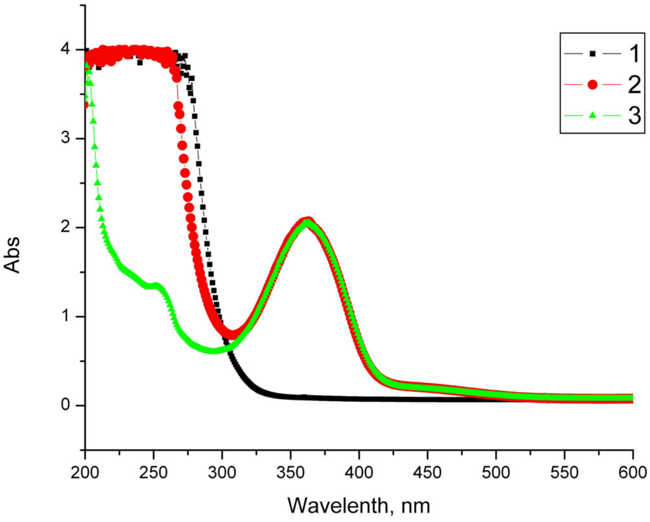


FIGURE 7 Comparative UV spectra of maleic anhydride (1), copolymer 2, and monomer 3

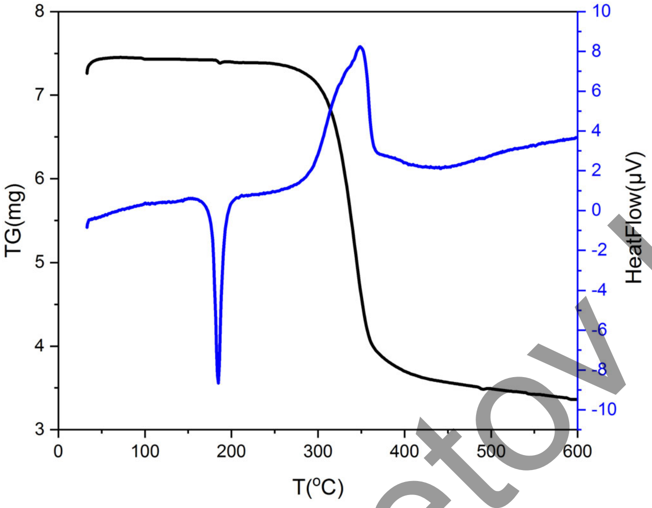


FIGURE 8 Thermal characteristics of copolymer 4

On the curve of the rate of mass loss in the temperature range 30 – 250°C, a slight change in speed is observed in the range from 7.25 to 3.2 mg/min. Starting from 300°C, a sharp increase in speed is observed and reaches its peak at 342°C, and the value of the speed at this temperature is 1.7 mg/min. Then comes a gradual decrease, and at 400°C the speed stabilizes, but in some places, there is a slight change at 475, 530, and 575°C.

An endothermic peak due to melting is observed on the DSC curve at a temperature of 180°C, while the substance mass does not change. Then, there is an exothermic effect at a temperature of ≈350°C, which is explained by the sample decomposition with the release of volatile products and mass loss.

4 | CONCLUSIONS

2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate containing the photochromic groups as first synthesized with the condensation reaction of 4-(2-hydroxyethyloxy)-4-cyanoazobenzene with cinnamoyl chloride, thus, its composition and structure were characterized by FTIR, UV–Vis, ^{1}H NMR spectroscopy, chromatography, XRD, and elemental analysis. X-ray crystallographic data for 4-(2-hydroxyethyloxy)-4-cyanoazobenzene have been deposited with the Cambridge Crystallographic Data Centre (2053997). The copolymer was synthesized by radical copolymerization of 2-(4-((4-isocyanophenyl)diazenyl)phenoxy)ethyl-3-phenylacrylate with maleic anhydride. The optical properties of the monomer and copolymer products were compared and the existence of *cis-trans* isomerization of azobenzene groups under the influence of UV radiation was detected. The thermal stability and phase transition temperature in a copolymer of 2-(4-((4-isocyanophenyl)diazenyl)phenoxy) ethyl-3-phenylacrylate with the maleic anhydride were determined using the DTA/DSC.

CONFLICT OF INTEREST

The authors declare no potential conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are openly available at the end of this article.

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