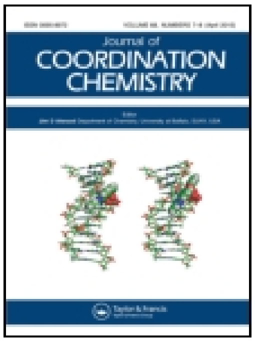

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Optically controlled distribution of *o*-quinonemethacrylate metal complexes in polymer materials

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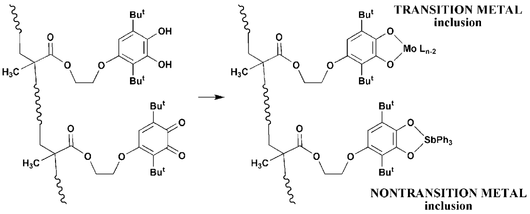

The complexation ability of photoreduction products of 2-(2,5-di-*tert*-butyl-3,4-dioxycyclohexa-1,5-dienyl)ethyl methacrylate in tetrahydrofuran (THF) solution and in its copolymer films with dimethacrylate and ethylene glycol monomethacrylate was studied. The product of photochemical reduction – catechol – reacts with $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, NH_4VO_3 and $(NH_4)_2Fe(SO_4)_2$ with the formation of corresponding metal complexes in solution and in the polymer bulk. The concentration of the molybdenum complexes in the polymer matrix was proportional to the exposure and holding time in solution of molybdate salt. Polymer film with optically controlled distribution of *o*-quinonemethacrylate molybdenum complexes was prepared. A film containing antimony and molybdenum complexes together in the polymer structure in predetermined ratio was obtained by sequential treatment of irradiated copolymer film (50% conversion) by THF solution of $SbPh_3$, and water solution of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$.

Keywords: *o*-Quinone; Catechol; Radical polymerization; Metal–polymer complexes; Thin films

1. Introduction

The search for synthetic ways and investigation of properties of metal-containing oligomers and polymers including polyoxometalates is one of the most intensively developing directions in modern coordination and materials chemistry [1–5]. Such interest is determined by

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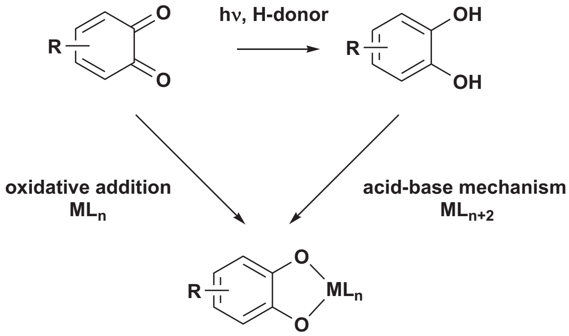
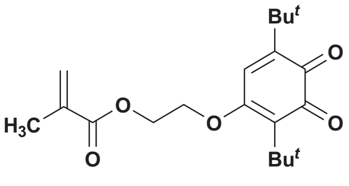
*Corresponding author. Email: aip@iomc.ras.ru a role they play in biological systems and industrial processes [6, 7]. One of the problems of synthesis of metal-containing polymers is the difficulties in preparation of heterobimetallic polymers with controllable content/distribution of metals. One way to solve this problem is copolymerization of ligands – monomers with different affinity to the metal. Another way is to provide a predetermined concentration gradient of ligands with different chelating ability in the bulk of material. This method should be realized by using materials containing photosensitive *o*-benzoquinones which can be reduced to catechols in the presence of H-donors [8, 9]. Both *o*-benzoquinones and catechols can be used as precursors in the synthesis of catecholato and paramagnetic *o*-semiquinonato transition and non-transition metal complexes [10, 11]. *o*-Quinones are active in oxidative addition and redox substitution of ligands. Corresponding catechols are weak acids and form metal complexes through acid–base mechanisms. The photolytic transformation of the ligand's redox state through a photoreduction of *o*-benzoquinones into catechols makes it possible to vary either the way of insertion or the quantity and the nature of metal (scheme 1).

Earlier [12], we prepared new *o*-benzoquinone functionalized by methacrylic group 2-(2,5-di-*tert*-butyl-3,4-dioxycyclohexa-1,5-dienyl)ethyl methacrylate (**QMEG**), active in polymerization (scheme 2), so that the photolytical setting of the ratio of the quinone and catechol moiety in the polymeric material can be used to introduce into the polymer a different amount of one metal or two different metals in a predetermined proportion.



Scheme 1. Synthetic ways to *o*-quinonato complexes.



(QMEG)

Scheme 2. 3,6-Di-*tert*-butyl-*o*-benzoquinone functionalized by methacrylic group.

2. Experimental

2.1. *Materials*

Solvents were purified according to standard procedures [13]. 2-(2,5-Di-*tert*-butyl-3,4-dioxycyclohexa-1,5-dieny)ethyl methacrylate (**QMEG**) and its polymer (**poly-QMEG**) were synthesized as described previously [13]. 4-Methyl-*N,N*-dimethylaniline (***p*-CH_3-DMA**) (Aldrich) was purified by distillation under reduced pressure. Monomethacrylate of ethyleneglycol (**MEG**) (Aldrich) and dimethacrylate of polyethyleneglycol-550 (**DMPEG**) (Aldrich) were used without purification. The initiator of polymerization was 2,2′-azobis[2-methylpropanenitrile] (**AIBN**) (Reachim) which was recrystallized from ethanol.

2.2. *Physical characterization*

^1H NMR spectra were recorded at frequencies of 200 MHz (Bruker Avance DPX-200), ^{13}C NMR spectra at 50 MHz, the solvent was $CDCl_3$. The infrared spectra of the compounds from 4000–400 cm^{-1} were recorded on a Specord M-80 spectrophotometer in nujol. The electronic absorption spectra were measured on a SF-56 (LOMO-spectrum) spectrometer at room temperature.

2.3. *Preparation of copolymer films*

Copolymer films were obtained by the following procedure: the composition for polymerization was prepared by the addition of **QMEG** (1.6 weight part (w.p.)) and **AIBN** (1 w.p.) to monomer mixture of **DMPEG** – **MEG** in the mass ratio of 1 : 1 (50 and 50 w.ps.). The mold consisting of two silicate glasses separated by Teflon spacer (h = 200 μm) was filled up by composition. The installation with composition was heated at 70°C during 20 h.

IR (cm^{-1}) of the copolymer film of composition: **QMEG** (1.6 w.p.) – **DMPEG** (50 w.p.) – **MEG** (50 w.p.) : 519 m, 652 w, 748 m, 818 w, 858 m, 947 m, 1030 w, 1246 m, 1272 m, 1300 m, 1320 w, 1351 m, 1360 w, 1386 m, 1396 w, 1454 s, 1485 s, 1551 m, 1624 m, 1639 m, 1686 m, 1724 s, 3470 s. Anal. Calcd for **QMEG** (1.6 w.p.) – **DMPEG** (50 w.p.) – **MEG** (50 w.p.) (%) : C, 56.25; H, 8.08; O, 35.67. Found (%) : C, 55.96; H, 7.97.

2.4. *2-(2,5-Di-tert-butyl-3,4-dihydroxybenzyl)ethyl methacrylate (H_2QMEG)*

QMEG solution (150 mg, 0.43 mmol) in tetrahydrofuran (THF) containing N,N-dimethylaniline (0.218 mL, 1.72 mmol, 4 eq) was irradiated in air (lamp KGM-24-150 with the light-focusing device, illuminance = 16 kLx) until formation of yellow solution. Catechol was obtained after recrystallization from hexane in the form of colorless crystals, m.p. 73–74°C. Anal. Calcd for $C_{20}H_{29}O_5$ (%) : C, 68.77; H, 8.31; O, 22.92. Found (%) : C, 68.72; H, 8.27. ^1H NMR (200 MHz, $CDCl_3$, δ, ppm) : 1.39 and 1.53 (s, both 9H; *t*-Bu), 1.96 (s, 3H; CH_3), 4.16 (t, J = 4.6 Hz, 2H; CH_2–O), 4.52 (t, J = 4.6 Hz, 2H, CH_2–O), 5.28 (w. s, 1H; –OH), 5.58 (s, 1H; H–CH=), 5.93 (w. s, 1H; –OH), 6.16 (s, 1H; H–CH=), 6.37 (s, 1H; C(q)–H). ^{13}C NMR (50 MHz, $CDCl_3$, δ, ppm) : 18.23 (CH_3), 29.64 and 31.54 (CH_3, *t*-Bu), 34.34 and 36.22 (C, *t*-Bu), 63.40 and 66.95 (CH_2–O), 125.96 and 136.11 (CH_2=C), 129.96 (C–H), 136.11, 149.77, 162.03, 167.41 (OC=O), C–OH). IR : ν (neat) 712, 731, 762, 789, 814, 867, 908, 947, 971, 1015, 1040, 1101, 1168, 1248, 1297, 1411, 1455, 1488, 1560, 1634, 1714, and 3449 cm^{-1}.

2.5. *Preparation of metal complex products of photoreduction of QMEG in solution and in polymer matrix*

Metal complexes with products of photoreduction of **QMEG** were prepared by treatment of irradiated **QMEG** THF solutions with aqueous solutions of ammonium metal salts [$(NH_4)_6Mo_7O_{24}$, NH_4VO_3 and $(NH_4)_2Fe(SO_4)_2$] until the organic phase becomes dark brown. **QMEG** (0.725×10^{-2} mol L^{-1}) solutions in THF containing *N,N*-dimethylaniline (2.9×10^{-2} mol L^{-1}) were irradiated in air (lamp KGM-24-150 with the light-focusing device, illuminance = 16 kLx), and then shaken with aqueous solutions of metal salts until the organic phase becomes dark brown. In order to obtain metal complexes with products of **QMEG** photoreduction in polymer bulk, the film was at first placed in a spectrophotometric cell $l = 0.1$ cm, filled with amine solution in THF (2.9×10^{-2} mol L^{-1}), and irradiated in air (lamp KGM-24-150, illuminance = 16 kLx). The changes in *o*-benzoquinone fragments concentration were observed spectrophotometrically by the change in intensity of the absorption band at $\lambda_{max} = 450$ nm. Then, the film was placed in the aqueous salt solution for 1 h.

To study the dependence of metal complex concentration on the irradiation time, the irradiated film was placed in an aqueous solution of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ for 5, 20, and 30 min. To study the dependence of the concentration of the formed complex from the intensity of the actinic radiation, the film was exposed through a photomask consisting of several "windows." The intensity of the actinic radiation passing through each photomask "window" in a fraction of the illuminance (16 kLx) is increased in the following order: 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0. After that, the irradiated film was placed in the aqueous salt solution for 1 h. The formation of the corresponding metal complexes was observed by UV–visible spectroscopy.

2.6. *Preparation of the bimetallic polymer film*

The polymer film was irradiated for the time required for half of photoreduction-benzoquinone fragments (conversion 50%). The red-brown color changed to light brown. Then, the film was treated with an aqueous salt solution of ammonium molybdate to form molybdenum-containing catecholate centers via reaction of catechol groups with molybdate salt through the acid–base mechanism. The color of the film turned brownish-black. The resulting film was placed in a deaerated THF solution of Ph_3Sb (it reacts with unphotoreduced *o*-benzoquinone fragments to give the corresponding triphenylantimony(V) catecholate), and then left in air [12]. All operations were accompanied by a change in color which was controlled spectrophotometrically. IR (cm^{-1}): 475 w, 520 m, 696 m, 729 w, 748 m, 856 m, 906 m, 950 m, 997 w, 1247 m, 1275 m, 1298 w, 1323 w, 1351 m, 1388 m, 1454 s, 1486 m, 1553 w, 1638 m, 1728 s, 3459 s.

3. Results and discussion

3.1. *Photoreduction of quinone-containing monomer and polymer*

It is known [8, 9] that visible light irradiation of benzoquinone solutions in the presence of a hydrogen donor (e.g. tertiary amines [14]) leads to the reaction of benzoquinone photoreduction to the corresponding colorless catechols. Irradiation of the **QMEG** solutions in

THF or toluene with visible light in the presence of **p-CH$_3$-DMA** leads to discoloration of the solutions (figure 1). One can observe that decrease in the intensity of the absorption band of the *o*-benzoquinone fragment in the region of $\lambda = 435$ nm is accompanied by growth of solution absorption in the UV region of the spectrum with a clear isosbestic point. The effective rate constant of photoreduction **QMEG** k_H is 25.7×10^{-4} s^{-1}. The "effective rate constant" is the observable constant which is calculated from the experimental data.

Previously, it was shown that sterically hindered *o*-benzoquinones photoreduced to the corresponding catechols [15–17]. According to NMR spectroscopy and elemental analysis, the product of **QMEG** photoreduction is catechol **H$_2$QMEG** (see Section 2.4) and, by analogy with other *o*-benzoquinone [15], the reaction can be described by scheme 3.

Obviously, the polymerization product of the quinone methacrylate – **poly-QMEG** – must also undergo photoreduction in the same conditions. To determine the comparative efficiency of this reaction in monomer and polymer solution – **poly-QMEG** (MM 32000) was synthesized by polymerization in mass [12]. Photoreduction of **poly-QMEG** in THF solution in the presence of **p-CH$_3$-DMA** was carried out under the same conditions as for the monomer, $k_H = 7.8 \times 10^{-4}$ s^{-1}. A factor of three reduction of k_H during the transition from monomer solution to the polymer solution is probably due to the difficulty of approach of the photoinduced amine molecules to quinone moieties in the polymer.

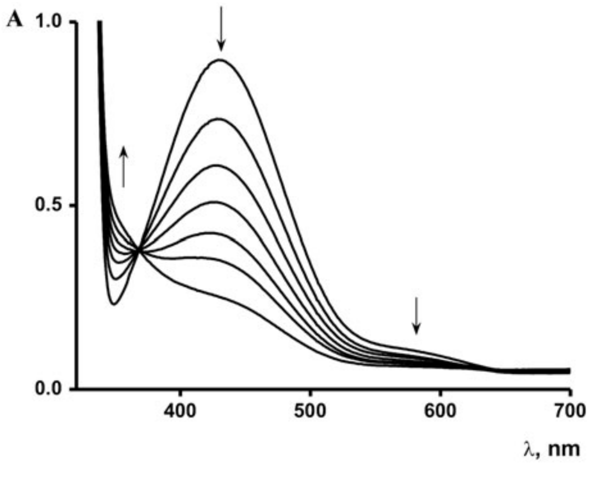
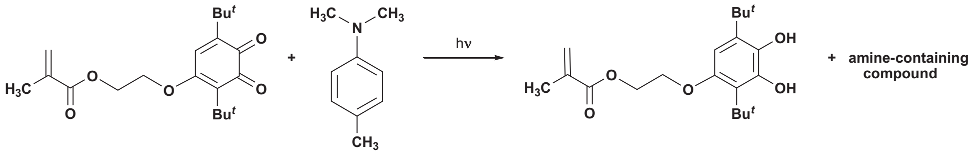


Figure 1. Spectral changes during irradiation of the **QMEG** solution (4.6×10^{-4} M) and **p-CH$_3$-DMA** (2.3×10^{-2} M) in THF (Ar, $\lambda > 500$ nm, the irradiation time of 280 s).



Scheme 3. Photoreduction of **QMEG** in the presence of **p-CH$_3$-DMA**.

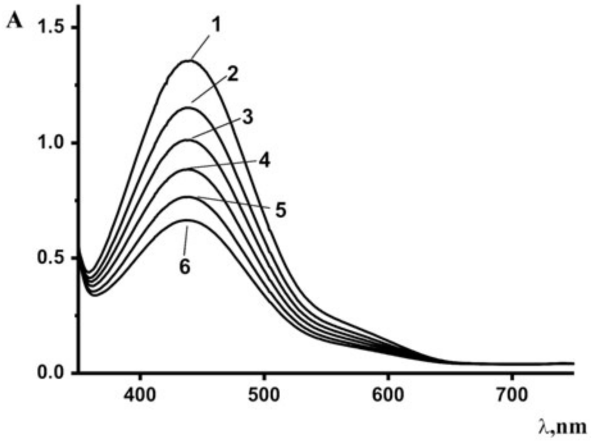


Figure 2. Spectral changes of the polymer film (h = 200 μm) prepared from the **QMEG** – **DMPEG** – **MEG** composition depending on the irradiation time (in min): (1) 0; (2) 3; (3) 6; (4) 9; (5) 12 and (6) 15.
Note: Irradiation in THF in the presence of ***p*-CH$_3$-DMA**, lamp KGM-24-150, air.

Attempts to obtain **poly-QMEG** in the form of polymer films did not lead to positive results because of the fragility of the polymer layer. At the same time, **QMEG** copolymerized with other mono- and dimethacrylates forming film materials [12]. In this study, dimethacrylate of polyethyleneglycol-550 (**DMPEG**) and monomethacrylate of ethylene glycol were taken as monomers to produce copolymers with **QMEG**. The polymer films with thickness of 200 μm were prepared from the composition containing 1.6 w.p. of **QMEG**, 50 w.p. of **DMPEG** and 50 w.p. of **MEG** by thermopolymerization. The film was placed in THF solution of ***p*-CH$_3$-DMA** and irradiated with visible light to convert part of *o*-benzoquinone fragments into catechol fragments. One can observe (figure 2) that irradiation of the film leads to decrease in the intensity of the absorption band of the *o*-benzoquinone fragment. The effective rate constant of *o*-quinone fragment photoreduction in polymer film k_H is 2.3×10^{-4} s^{-1}.

3.2. *Preparation of metal complexes and metal polymers*

Treatment of toluene solution of 3,5-di-*tert*-butyl-catechol with aqueous metal salt solutions leads to formation of the corresponding catecholato metal complexes in the organic phase [18, 19]. Catechols in polymer are also able to form complexes with metals [20–22]. Treatment of the **QMEG** photoreduction product solution in toluene with an aqueous solution of $(NH_4)_6Mo_7O_{24}$, or NH_4VO_3, or $(NH_4)_2Fe(SO_4)_2$ leads to coloration of the organic phase. Electronic absorption spectra of the **QMEG** toluene solution after irradiation (curve 1) and after treatment with aqueous solutions of $(NH_4)_6Mo_7O_{24}$ (curve 2), NH_4VO_3 (curve 3) and $(NH_4)_2Fe(SO_4)_2$ (curve 4) are represented in figure 3(a).

Treatment of irradiated **QMEG** solution with aqueous solutions of metal salts gives a set of bands in the electronic absorption spectra. This, by analogy, can be attributed to absorption of *o*-quinonato complexes of the corresponding metal catecholates [19].

The results of similar experiments with **QMEG** – **DMPEG** – **MEG** copolymer films are illustrated in figure 3(b). The irradiated films were placed in an aqueous salt solution and

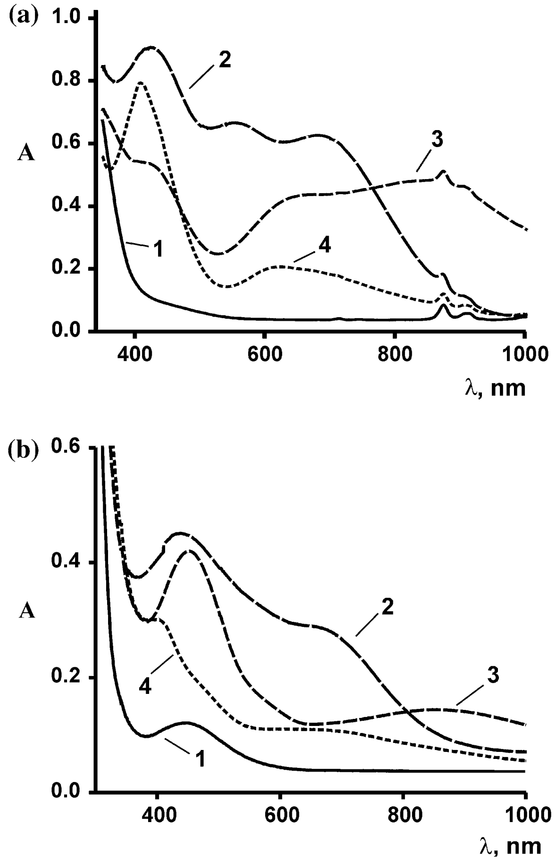


Figure 3. Electronic absorption spectra of **QMEG** solution in toluene (a) and the polymer film (h = 200 μm), a copolymer **QMEG** – **DMPEG** – **MEG** (b) after exposure (in the presence of ***p*-CH$_3$-DMA**, lamp KGM-24-150, air) (1); after treatment with aqueous solutions of the irradiated samples $(NH_4)_6Mo_7O_{24}$ (2), NH_4VO_3 (3), $(NH_4)_2Fe(SO_4)_2$ (4).

kept for 1 h. The comparison of the absorption spectra of **QMEG** metal complexes obtained in toluene and in **QMEG** copolymer films shows that they are not identical to each other. The reason for this lies in the fact that several complexes may be formed by reaction of catechol with metal salt. These complexes can differ by number of oxygens and the presence in the coordination sphere of molecules of solvent and amine, and also catechol form of reduced benzoquinone fragments. Apparently, the ratio between the various structures of metal complexes in solution and in the polymer film should be different. The concentration of metal complexes in polymer film also depends on the time of treatment of the irradiated polymer film by aqueous solution of metal salt. The dynamics of this process for reaction with ammonium molybdate are shown in figure 4.

Figure S1(a) of Electronic Supplementary information shows a photo of the polymer material after exposing through the photomask. The intensity of the actinic radiation passing through each "window" of the template (from the upper left) is increased as follows: 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0. It is seen that the degree of "burnout" of the coloration of the polymer increases with increasing radiation intensity, which is associated with the presence

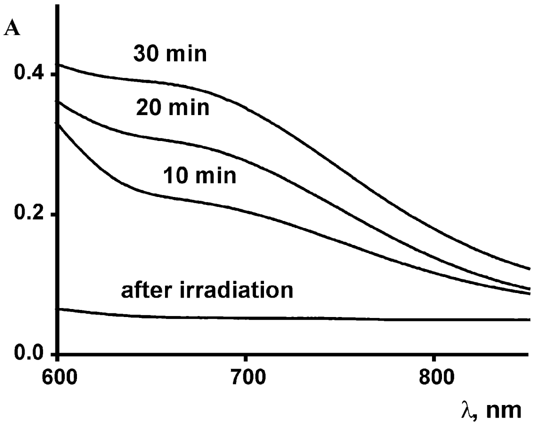


Figure 4. The dependence of spectral characteristics of the exposed polymer film (30 min) from the processing time in an aqueous solution of $(NH_4)_6Mo_7O_{24}$.

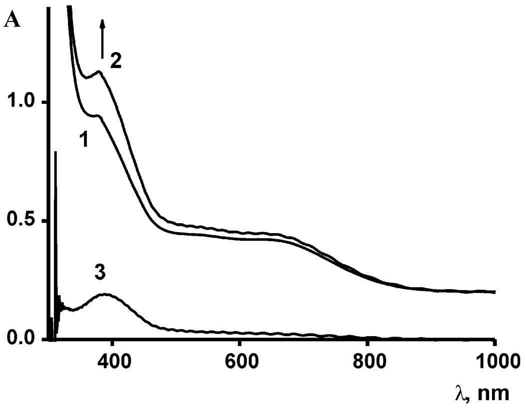
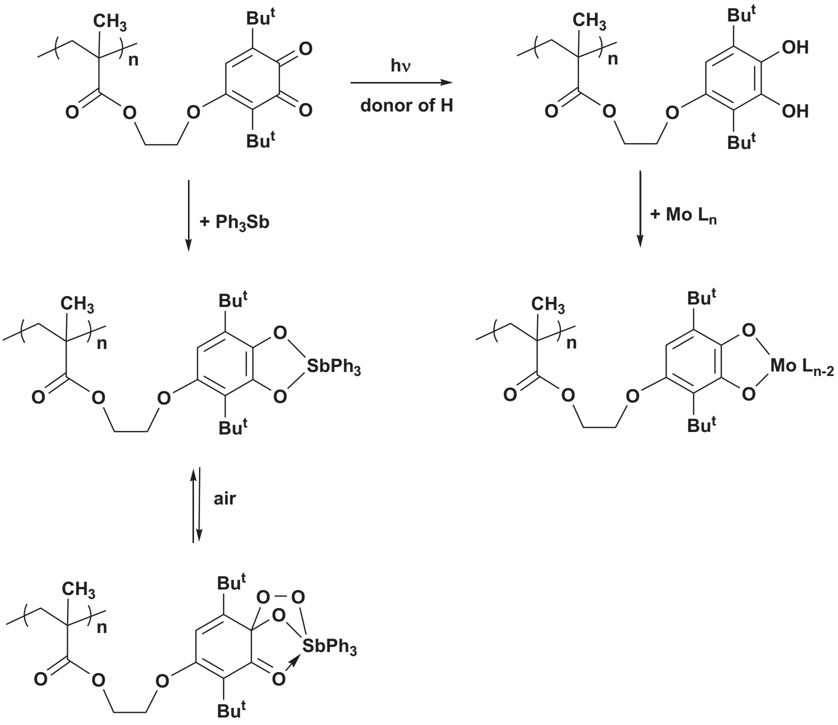


Figure 5. Absorption spectra of the exposed and processed with an aqueous solution of $(NH_4)_6Mo_7O_{24}$ copolymer film composition **QMEG** – **DMPEG** – **MEG** (h = 200 μm): (1) after the treatment with Ph_3Sb; (2) after exposure of the material to air, (3) "calculated" spectrum obtained by subtracting spectrum 1 from spectrum 2.

of *o*-benzoquinone fragments. Figure S1(b) of Electronic Supplementary information shows a photo of the irradiated layer after its treatment in solution of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$. With increasing actinic radiation doses, consistent increase in the optical density takes place that corresponds to increase in the metal complex content in irradiated plot of the polymer layer.

The reaction of *o*-benzoquinones with Ph_3Sb with the formation of antimony(V) catecholate complex [12] can be used as a qualitative reaction on *o*-benzoquinone. Some of these complexes are able to bind molecular oxygen in a reversible manner with change of color [23, 24]. Previously, we have shown that *o*-benzoquinone fragments in the **QMEG** – **DMPEG** – **MEG** copolymer film are also capable of forming antimony complexes by reaction with Ph_3Sb [12]. This makes possible to obtain the polymer film containing both antimony and other metal complexes in the photolytically determined ratio. Practically,



Scheme 4. Processes realized in copolymer film.

QMEG – **DMPEG** – **MEG** copolymer film irradiated during the time required for half of the photoreduction of *o*-benzoquinone fragments was treated with an aqueous salt solution of ammonium molybdate. Then, it was placed into deaerated THF solution of Ph_3Sb, and further exposed to air. All operations were accompanied by a change in color which was monitored spectrophotometrically. The absorption spectrum of the film after the treatment with Ph_3Sb solution (range 1) is shown in figure 5. It contains the absorption band of molybdenum complex at 650–700 nm. Exposure of the film to air leads to increase of absorption in the blue and UV-spectral regions (spectrum 2). Subtraction of the spectrum 1 from the spectrum 2 gives the spectrum 3 containing a single absorption band with a maximum of 380–390 nm, which corresponds to the absorption spectrum of the triphenylantimony spiro endoperoxide complex in the polymer. The related triphenylantimony(V) 4-methoxy-3,6-di-*tert*-butyl-catecholate and 4,5-dimethoxy-3,6-di-*tert*-butyl-catecholate react with molecular oxygen with formation of the corresponding spiro endoperoxides, and this spiro endoperoxide formation is accompanied by the appearance of similar bands in electronic absorption spectra with maxima at 383 and 372 nm, respectively [24]. The heating and deaeration of spiro endoperoxide solutions leads to disappearance of these bands and formation of initial triphenylantimony(V) catecholates [24]. After heating the film under an inert atmosphere, the absorption spectrum 2 of the film changes in the short wavelength region and transforms into spectrum 1. After repeated exposure of the film to air in the polymer, absorption spectrum 1 transforms back to spectrum 2. These results demonstrate that triphenylantimony(V) catecholate moieties in our polymer film are also able to bind molecular oxygen in a reversible manner.

These results demonstrate that the copolymer film contains catecholate complexes of triphenylantimony and molybdenum complexes. Processes realized in copolymer film are shown in scheme 4.

4. Conclusion

Insertion of two metals – antimony and molybdenum – into one region of the **QMEG** – **DMPEG** – **MEG** copolymer film (1.6 : 50 : 50 w.p.) was realized by partial photoreduction of *o*-benzoquinone fragments and different mechanisms of complexation for *o*-benzoquinone and its photoreduction products, showing the possibility for preparation of polymer material with optically controlled distribution of metals.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Supplemental data

Supplemental data for this article can be accessed http://dx.doi.org/10.1080/00958972.2015.1089355.

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