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Journal of Photochemistry and Photobiology A: Chemistry 116 (1998) 251–256

Journal of Photochemistry and Photobiology A:Chemistry

Polymeric photosensitizers
2. Photosensitized oxidation of phenol in aqueous solution

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Received 20 March 1998; accepted 26 May 1998

Abstract

Poly(sodium styrenesulfonate-*co*-vinylbenzyl chloride) with rose bengal chromophores covalently attached to the polymer chain (PSSS-VBC/RB) has been successfully used as a photosensitizer for oxidation of phenol in an aqueous solution. *p*-Benzoquinone was identified as a primary photochemical product. The quantum yields of photooxidation of phenol was found to be strongly dependent on pH of aqueous solution.

Keywords: Polymeric photosensitizer; Phenol; Aqueous solution

1. Introduction

Photosensitized oxidation of organic compounds has been a subject of intensive research. One approach which was taken in these studies was oriented towards a development of the method for synthesis of fine chemicals. The recent interest in the field is related mainly to the photocatalytic degradation of pollutants [1–16].

A variety of photosensitizers has been developed over the years. Recently, the sensitizers belonging to one of two major groups have been mostly studied; semiconductors [1–14] and dyes [13,15–23] [24,25,30,31]. Semiconductors, mainly TiO_2 dispersions, have been used for the photocatalytic degradation of pollutants [1–14]. It is well known that photogenerated and separated electrons and holes in such systems can induce redox reactions [1]. Dye-sensitized oxidation processes involve participation of the reactive oxygen species, mainly, singlet oxygen [13,15–25,30,31].

Rose bengal, a xantene dye, has been shown to act as very efficient generator for singlet oxygen [17,22]. Rose bengal photosensitized oxidation of organic compounds can be carried out in homogenous [16], microheterogenous [26–30] as well as in heterogeneous systems [20,21]. Recently we developed a new homogenous polymeric photosensitizer, poly (sodium styrenesulfonate-*co*-vinylbenzyl chloride) with covalently attached rose bengal chromophores (PSSS-VBC/RB) and demonstrated its ability for efficient formation of singlet oxygen [32]. This paper presents the application of PSSS-VBC/RB as a photosensitizer for oxidation of phenol in aqueous solution.

2. Experimental part

2.1. Materials

Synthesis and properties of PSSS-VBC/RB were described earlier [32]. The polymer contained 75 mol% of sodium styrene sulfonate (SSS) and 25 mol% of vinylbenzyl chloride (VBC). The content of RB attached to the polymer chain was estimated to be 55 wt.%.

Phenol (PhOH) (POCh, Gliwice, Poland, pure) was purified by sublimation. *p*-Benzoquinone (BQ) (POCh, Gliwice, Poland, pure) was crystallized from hexane. Hydroquinone (HQ) was crystallized from water. $K[Cr(NH_3)_2(SCN)_4]$ was obtained from the commercial Reinecke's salt and purified according to the procedure described in the literature [33].

Aqueous solutions of the reactants were prepared using twice distilled water. The pH of the solutions was adjusted by using the buffer solutions (H_3BO_3–$Na_2B_4O_7 \cdot 10\ H_2O$, NaH_2PO_4–K_2HPO_4) or NaOH.

Hexane (Merck, spectral grade), carbon tetrachloride (POCh Gliwice, spectral grade), rose bengal (RB, Aldrich, certified grade, 92 wt.%), borax ($Na_2BO_7 \cdot 10\ H_2O$), NaH_2PO_4, K_2HPO_4, boric acid and sulfuric acid were used without further purification.

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PII S1010-6030(98)00305-0

2.2. Apparatus and procedures

2.2.1. Irradiation of the samples

The samples were irradiated using a medium pressure mercury lamp (ASH400) and glass filter with a cut-off at $\lambda > 535$ nm. It has been found that the incident radiation consists only of two mercury lines at 546 nm and 578 nm (see Fig. 1). The radiation at $\lambda = 578$ nm is hardly absorbed by RB chromophores and can be neglected in further discussion. The photochemical experiments were carried out as follows. In a typical experiment, 3 cm^3 of aqueous solution of phenol (2.9×10^{-3} mol dm^{-3}) and PSSS-VBC/RB (21.5 mg dm^{-3}) was placed in the reaction vessel. Oxygen gas was bubbled continuously through the solution during irradiation.

The intensities of the light absorbed by the samples were determined using the potassium reineckate actinometer $K[Cr(NH_3)_2(SCN)_4]$ [33]. The concentration of potassium Reinecke's salt was 1.24×10^{-2} mol dm^{-3} at $5.3 < pH < 5.5$ and the solution was bubbled with argon during irradiation.

The mean intensity of the light absorbed by PSSS-VBC/RB, $\bar{I}_{abs}$, was calculated according to Eq. (1).

$$\bar{I}_{abs} = I_{546} \int_{\lambda} T(\lambda) F(\lambda) (1 - 10^{-A_{RB}(\lambda)}) d\lambda \qquad (1)$$

where $T(\lambda)$ is transmittance of the filter shown in Fig. 1, $A_{RB}(\lambda)$ is the absorbance of PSSS-VBC/RB and $F(\lambda)$ is the spectral distribution of the light emitted by the lamp given as $I(\lambda) = I_{546} F(\lambda)$ (see Fig. 1).

In order to determine the intensity of the light emitted by the lamp the actinometer solution was placed in the reaction cell and exposed to radiation while bubbling with argon. The rate of reaction in the actinometer solution, V_r, was measured and can be expressed as follows:

$$V_r = I_{546} \int_{\lambda} \phi_R(\lambda) T(\lambda) F(\lambda) (1 - 10^{-A_R(\lambda)}) d\lambda \qquad (2)$$

where $\phi_R(\lambda)$ is quantum yield of the reaction, $A_R(\lambda)$ is the absorbance of Reinecke salt. It was found that, under these experimental conditions, the mean intensity of the light absorbed by the PSSS-VBC/RB solution was $\bar{I}_{abs} = (4.66 \pm 0.08) \times 10^{-6}$ einstein dm^{-3} s^{-1}.

UV–Vis absorption spectra of the samples were recorded at room temperature using a Hewlett-Packard 8452A diode array spectrophotometer.

IR spectra of the samples were recorded at room temperature on a Bruker IFS 48 FT spectrophotometer. The samples were prepared by extraction of the organic material from irradiated aqueous solution with CCl_4.

2.2.2. Gas chromatography–mass spectrometry analyses

The analyses of the systems during irradiation were performed by means of Hewlett-Packard HP 6890 Series GC System gas chromatograph equipped with a flame ionization detector (FID) and DB-5 capillary column (30 m, 0.32 mm ID, 0.25 mm film thickness). The samples for analyses were prepared by extraction of irradiated aqueous solutions with hexane. Typically 1 cm^3 of hexane and 0.25 cm^3 of H_2SO_4 (5 mol dm^{-3}) were added to 2 cm^3 of the aqueous solutions and shaken for about 15 min. The organic phase was separated and dried over $CaCl_2$.

The products formed upon irradiation were identified using Hewlett-Packard 5890 GC/MS instrument equipped with a HP-1 capillary column (12 m, 0.20 mm ID, 0.33 mm film thickness).

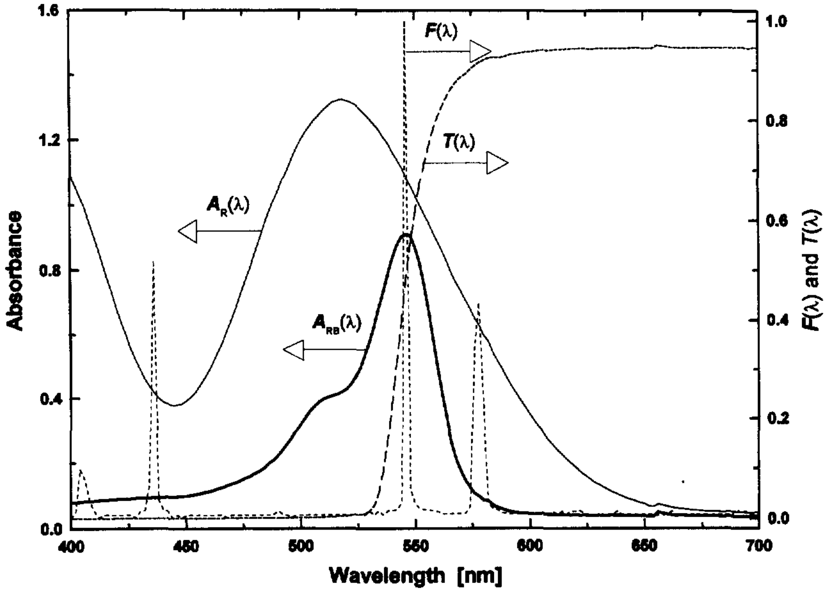

Fig. 1. Spectral characteristics of the system studied; $A_{RB}(\lambda)$—absorption band of RB chromophores attached to PSSS-VBC polymer in the visible spectral region ($c = 21.5$ mg dm^{-3}), $A_R(\lambda)$—absorption band of actinometer, $F(\lambda)$—the spectral distribution of the light emitted by the lamp, $T(\lambda)$—transmittance of the cut-off filter.

3. Results and discussion

3.1. PSSS-VBC/RB photosensitized oxidation of phenol in aqueous solution

Oxygen-saturated aqueous solutions of phenol and PSSS-VBC/RB adjusted to defined pH were irradiated with light absorbed only by RB chromophores attached to the polymer chain ($\lambda > 535$ nm). It was observed that irradiation resulted in decrease in concentration of phenol. The measurements of the UV–Vis absorption of the system (pH = 7.2) demonstrated that the irradiation leads to a decrease in the intensity of absorption band at $\lambda = 270$ nm, characteristic for phenol [34], and appearance of a new absorption band with a maximum at $\lambda = 246$ nm (see Fig. 2). The reaction was also followed by the GC, GC/MS and IR analyses. It has been shown that the main primary product of photosensitized oxidation of phenol is *p*-benzoquinone (BQ). This is consistent with the observation of the mentioned above changes in the UV spectrum of irradiated phenol; BQ has an absorption band with the maximum at 246 nm [41]. The GC analyses have indicated that a second minor product was present in the irradiated solution (phenol + PSSS-VBC/RB). The product was identified by GC–MS as hydroquinone (HQ). HQ was suggested to be the second, minor primary product of photooxidation of phenol sensitized by rose bengal in alkaline solution [16]. This product, however, was not present when photooxidation of phenol was carried out in acidic aqueous solutions ($2 < \text{pH} < 4$) [37]. Thus one can consider that hydroquinone can be also formed as a secondary product of dark reaction occurring between BQ and alkali [38]. (BQ is known to participate in the complicated dark reactions in aqueous alkaline solution [16,38,40]). BQ was reported to be the only product during photooxidation of phenol in acidic solutions (pH = 2, 3 or 4) [13,37].

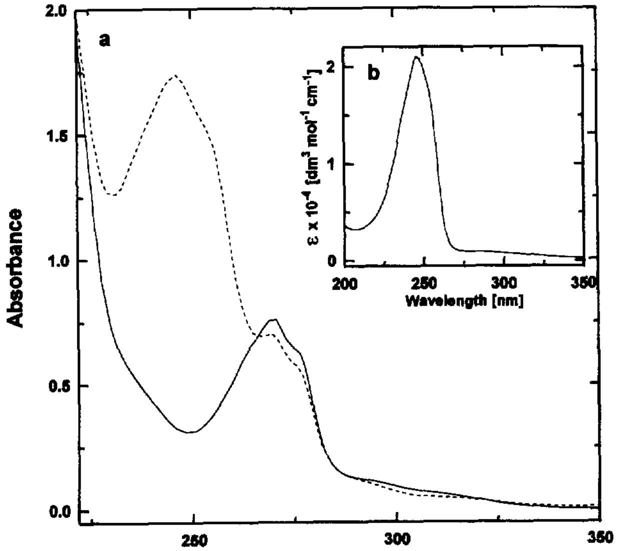

Fig. 2. a) Electronic absorption spectra in the UV spectral region for an aqueous solution of PSSS-VBC/RB + phenol before irradiation (—) and after irradiation for 3 h (- - -), the light being absorbed only by RB chromophores ($\lambda > 535$ nm, neutral pH); b) UV absorption spectrum of aqueous solution of *p*-benzoquinone.

3.2. The pH effect on the rate and the quantum yield of degradation of phenol

The kinetics of photooxidation of phenol is strongly dependent on the pH of the irradiated solution. Fig. 3 shows the changes in concentration of phenol observed during irradiation of PSSS-VBC/RB + phenol in aqueous solutions of various pHs.

The rate of the process increases with an increase in pH (see Table 1). The experiments described in this paper were restricted to the pH > 6 because in highly acidic solutions the photosensitizer used (RB chromophore) does not absorb the light from the spectral region chosen and it is not efficiently generating singlet oxygen.

The quantum yield of photooxidation of phenol, $\Phi_{\text{-PhOH}}$, and the efficiency of the photooxidation, E, can be calculated using the Eqs. (3) and (4), respectively:

$$\Phi_{-\text{PhOH}} = \frac{V_r^0}{I_{\text{abs}}} \tag{3}$$

and

$$E = \frac{\Phi_{-\text{PhOH}}}{\phi_{^1\text{O}_2}} \tag{4}$$

where: V_r^0 is the initial rate of reaction (consumption of phenol) determined at conversion lower than 10% to avoid any possible interference from the products formed and $\phi_{^1\text{O}_2}$ is the quantum yield of singlet oxygen formation in the system, which was determined earlier as equal to 0.73 ± 0.05 [32].

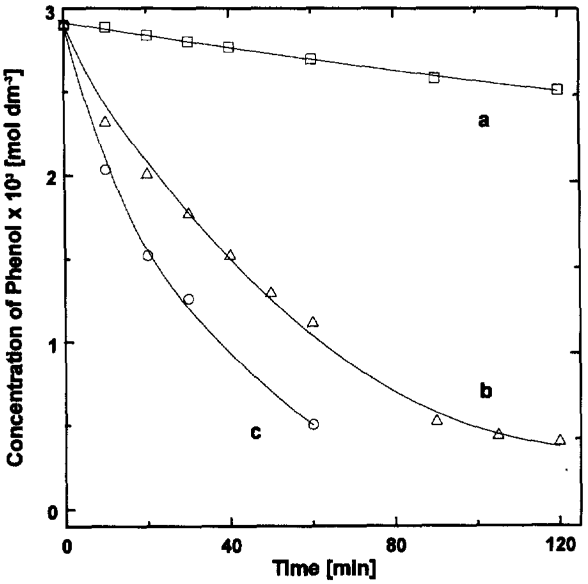

Fig. 3. Changes in the concentration of phenol on irradiation time of aqueous solutions of PSSS-VBC/RB + phenol at various pH ($\lambda > 535$ nm): (a) pH = 7.2; (b) pH = 10.3; (c) pH = 11.3.

Table 1
Experimental data for photosensitized oxidation of phenol

pH	α	[PhOH] × 10^3 [mol dm^{-3}]	[PhO^-] × 10^3 [mol dm^{-3}]	V_r^0 × 10^7 [mol dm^{-3} s^{-1}]	$\Phi_{\text{-PhOH}}$ (±7%)	E (±9%)
6.38	2.4×10^{-4}	2.8993	0.0007	0.070	0.0015	0.002
7.20	0.0016	2.8953	0.0047	0.231	0.005	0.007
8.16	0.0146	2.8577	0.0423	0.697	0.015	0.020
8.50	0.0313	2.8091	0.0909	0.362	0.008	0.011
8.81	0.0620	2.2720	0.1797	1.790	0.038	0.053
9.25	0.1540	2.4535	0.4465	2.522	0.054	0.074
9.57	0.2755	2.1012	0.7988	4.105	0.088	0.121
10.30	0.6712	0.9534	1.9466	9.750	0.212	0.290
11.30	0.9533	0.1354	2.7646	14.390	0.309	0.423

The initial rates of reaction, V_r^0, were determined from the dependencies of concentration of phenol on irradiation time for early stage of the reaction. The results are listed in Table 1.

Fig. 4 shows that the quantum yield of photooxidation of phenol increases with increase of pH of irradiated solution. The value of quantum yield found for pH = 10.3, $\Phi_{\text{-PhOH}} = 0.212 \pm 0.015$, compares well with the value of 0.29 obtained by Okamoto et al. [16] at the same pH, when free rose bengal was used as a photosensitizer. This indicates that the polymer present in the system does not lead to the substantial inhibition of the oxidation of phenol.

The pH effect on the kinetics of photooxidation of phenol can be explained considering the dissociation of phenol. A degree of dissociation (α) at any given pH can be calculated as follows:

$$\alpha = \frac{[PhO^-]}{[PhO^-]+[PhOH]} = 1/(1+10^{(pK_\alpha - pH)}) \qquad (5)$$

where pH is the measured value and $pK_\alpha = 9.99$ [41].

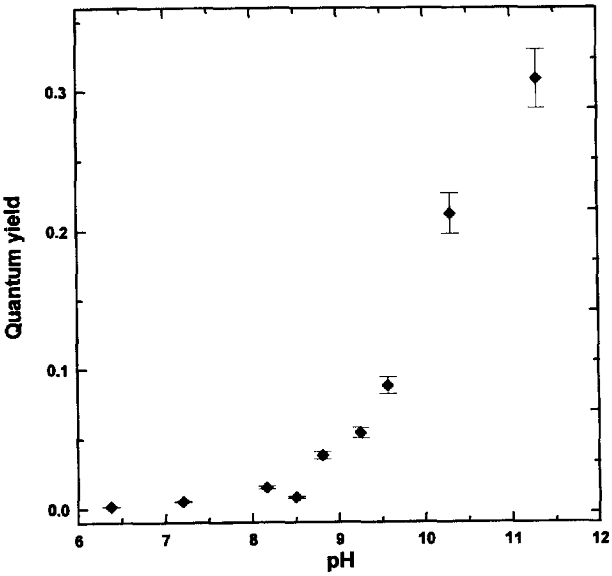

Fig. 4. Dependence of the quantum yield of photooxidation of phenol on the pH of the aqueous solution of PSSS-VBC/RB.

The calculated values of α and the concentrations of PhOH and PhO^- in solutions at the various pH used in our experiments are collected in Table 1.

Taking into account the experimental results and the literature data [13,16,18,35,36,39] the photosensitized by PSSS-VBC/RB oxidation of PhOH in an aqueous solution can be described as follows:

Process	*Rate*	
PSSS-VBC/$^1RB_0 + h\nu \rightarrow$ PSSS-VBC/$^1RB^*$	$\bar{I}_{abs}$	(6)
PSSS-VBC/$^1RB^*$ $\rightarrow$ PSSS-VBC/$^3RB^*$	$k_{ISC}[^1RB^*]$	(7)
PSSS-VBC/$^3RB^* + {}^3O_2 \rightarrow$ PSSS-VBC/$^1RB_0 + {}^1O^*_2$	$k_q[^3RB^*][O_2]$	(8)
$^1O_2^* \rightarrow {}^3O^*_2$	$k_d[^1O^*_2]$	(9)
$PhOH + {}^1O^*_2 \rightarrow BQ$	$k_1[^1O^*_2][PhOH]$	(10)
$PhOH + {}^1O^*_2 \rightarrow PhOH + {}^3O_2$	$k_q^{PhOH}[^1O_2^*][PhOH]$	(11)
$PhO^- + {}^1O^*_2 \rightarrow BQ$	$k_2[^1O^*_2][PhO^-]$	(12)

The scheme involves the process of singlet oxygen formation by quenching of RB chromophores (in their triplet states) by oxygen in the ground state (Eq. (7)). The generation of singlet oxygen by PSSS-VBC/RB has been demonstrated previously [32]. The mechanism of an interaction between singlet oxygen and phenol has been the subject of considerable interest [13,16,35,36,39]. It has been established that the process involves both physical deactivation of singlet oxygen by phenol and chemical reaction between these two molecules. The chemical reaction occurs in several stages. It is initiated by the formation of a charge-transfer complex between singlet oxygen and the molecule of phenol in a ground electronic state. This facilitates the process of complete electron transfer from phenol to singlet oxygen resulting in the formation of a phenoxy radical. The reaction between the phenoxy radical and oxygen leads to the formation of quinone.

The observed increase in the rate of consumption of phenol at higher pH can be explained considering possible higher reactivity of PhO^- than PhOH towards singlet oxygen. The physical quenching of singlet oxygen by phenol should also be considered.

The rate of photooxidation of phenol can be expressed as follows:

$$V_r = k_1[{}^1O_2^*][PhOH] + k_2[{}^1O_2^*][PhO^-] \quad (13)$$

where

$$[{}^1O_2^*] = \frac{\bar{I}_{abs}\phi_{{}^1O_2}}{k_d + (k_1 + k_q^{PhOH})[PhOH] + k_2[PhO^-]} \quad (14)$$

In order to determine the rates constant k_1 and k_q^{PhOH} the photosensitized oxidation of phenol was studied at various initial concentrations in acidic solution of fixed pH ($pH = 6.38$). At the chosen pH, the value of the degree of dissociation is very low, $\alpha = 0.00024$, and the participation of PhO^- in the overall reaction can be neglected.

Thus the equation describing the rate of photooxidation of phenol in acidic solution can be reduced to the form:

$$V_r = \bar{I}_{abs}\phi_{{}^1O_2}\frac{k_1[PhOH]}{k_d + (k_1 + k_q^{PhOH})[PhOH]} \quad (15)$$

The modification of that equation gives the dependence of $1/V_r$ on $1/[PhOH]$:

$$1/V_r = \frac{k_d}{\bar{I}_{abs}\phi_{{}^1O_2}k_1} \cdot \frac{1}{[PhOH]} + \frac{k_1 + k_q^{PhOH}}{\bar{I}_{abs}\phi_{{}^1O_2}k_1} \quad (16)$$

Fig. 5 shows the dependence of $1/V_r^0$ on $1/[PhOH]$ based on the experimentally found rates of phenol consumption at fixed pH value ($pH = 6.38$) and various initial concentration of phenol.

From the y axis intercept and tangent of the straight line (Fig. 5), and by using the experimental values for $\bar{I}_{abs} = 4.66 \times 10^{-6}$ einstein $dm^{-3}\ s^{-1}$, $\phi_{{}^1O_2} = 0.73$ and $k_d = 4.4 \times 10^5\ s^{-1}$ [19], it was found that

$$k_1 = (1.05 \pm 0.05) \times 10^6\ dm^3\ mol^{-1}\ s^{-1}$$

and

$$k_q^{PhOH} = (3.45 \pm 0.26) \times 10^8\ dm^3\ mol^{-1}\ s^{-1}$$

The rate constant k_2 was determined as a mean value obtained from the measurements of the rate of photosensitized oxidation of phenol at high pH ($10.3 < pH < 11.3$) using the Eq. (13);

$$k_2 = (1.40 \pm 0.20) \times 10^8\ dm^3\ mol^{-1}\ s^{-1}$$

The values of k_1 and k_2 compare quite well with the literature values [42]. Unfortunately we have not been able to find in the literature the value of k_q^{PhOH} for phenol in water. The values of the rate of physical quenching of ${}^1O_2$ by α-tocopherol in organic solvents are in the range of 1×10^8–$6 \times 10^8\ dm^3\ mol^{-1}\ s^{-1}$ [19].

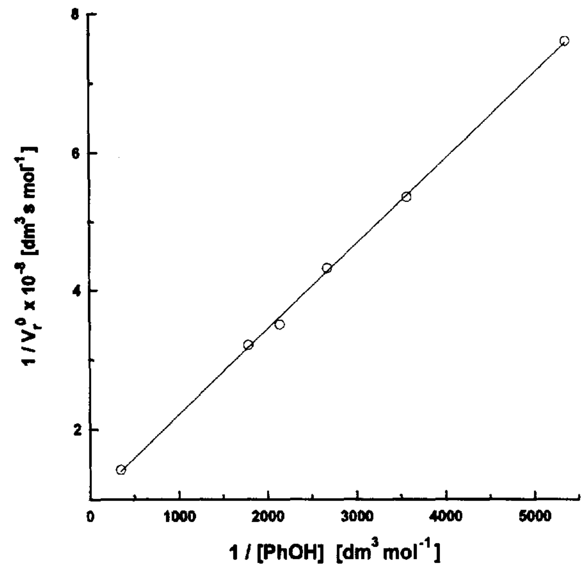

Fig. 5. Dependence of $1/V_r^0$ on $1/[PhOH]$ at $pH = 6.38$ (V_r^0 is the initial rate of consumption of phenol).

3.3. Bleaching of RB chromophores

It has been observed that under the experimental conditions RB is also consumed. That process, however, was not introduced in the above reaction scheme because it is considerably slower than the oxidation of phenol. The decrease in the concentration of RB chromophores was found to occur with the rate of 6×10^{-7} mol $dm^{-3}\ h^{-1}$; so at $pH = 10.3$, the concentration of phenol drops by half while the concentration of RB polymeric chromophores decreases only to 94%.

3.4. Separation of photosensitizer

The possibility of separation of the polymeric photosensitizer from reactants after completion of reaction was also investigated. The following ways of separation seem to be the most promising:

1. column chromatography
2. dialysis
3. filtration

In the first case, chromatography column (25 cm) was filled with silica gel (60 mesh). The aqueous solution of PSSS-VBC/RB + reactants were introduced to the column, and water or a mixture of water–methanol (50/50 v/v%) was used as the eluent. The polymer was adsorbed on the silica gel of the column while the low-molecular-weight substances were quantitatively eluted.

In a second approach, the aqueous solution of PSSS-VBC/RB + reactants was dialysed against deionized water. The polymer was recovered by freeze drying.

In the last case, the filtration was carried out through a layer of aluminium oxide (1 cm thick). The polymer was readily adsorbed. Unfortunately, it seems that the polymer is adsorbed permanently to Al_2O_3. The attempts to recover the polymer by extraction with various solvents (at ambient or higher temperature) were not successful.

4. Conclusions

It has been shown that PSSS-VBC/RB act as efficient photosensitizer in reaction of photodegradation of phenol in aqueous solutions. The reaction occurs with participation of singlet oxygen. 1,4-Benzoquinone was identified as main primary photochemical product. The quantum yields of the reaction are pH dependent and they are considerably higher in alkaline than in a neutral solution. That fact can be explained by considering both the higher reactivity of PhO$^-$ than PhOH with singlet oxygen and the physical quenching of singlet oxygen by phenol. The polymeric photosensitizer can be easily separated from the low-molecular-weight reactants and reuse in the new cycle of the reaction.

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