

Effect of light sources on the phenol degradation using Pt/TiO₂ photocatalysts immobilized on glass fibres

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Abstract

The layer of Pt/TiO₂ (1 mol.% Pt:Ti) was immobilized by sol–gel technique on the glass fibres and the prepared material was utilized for the photocatalytic phenol degradation using four different 15 W emission lamps as the illumination sources. The experimental conditions of the photocatalytic process were optimized in the relation to phenol concentration and oxygen flow. The apparent quantum yields of phenol degradation were calculated for radiation sources applied. © 1998 Elsevier Science S.A.

Keywords: Pt/TiO₂; Photocatalysis; Phenol degradation

1. Introduction

The irradiation of active titanium dioxide photocatalysts in aqueous systems under the aerobic conditions leads to the formations of extremely reactive species, i.e., TiO₂(h⁺), O₂^{•-}, [•]HO₂, [•]OH, H₂O₂, tetroxide [1–5], which effectively non-selectively destroy organic pollutants dissolved in water [3,6–10]. As phenol and phenol derivatives are wide-spread pollutants of the aquatic environment, the great attention was previously focused on the study of their photocatalytic decomposition [11–22]. Due to the unsuitable properties of TiO₂ suspensions for the large scale technical applications in the wastewater treatment, the alternative methods of TiO₂ photocatalysts immobilization or preparation on a suitable support were investigated [8–10,23–29]. On the basis of our previous work [30], using sol–gel method Pt/TiO₂ (1 mol.% Pt:Ti) photocatalysts supported on glass fibres was prepared and applied for the photocatalytic phenol degradation with the main aim to investigate the effect of spectral characteristics of 15 W standard emission lamps on the apparent quantum yield of phenol degradation.

2. Experimental

2.1. Materials

Titanium(IV) tetrabutoxide, Ti[O(CH₂)₃CH₃]₄, 99%, and ethyl acetoacetate, 99% were purchased from Aldrich.

Methanol (quality for gas chromatography) and ethanol (spectroscopic grade) were obtained from Lachema (Czech Republic), as well as nitric acid and phosphoric acid. Ethanol used for TiO₂ sol preparation was dried using sodium, and then distilled.

Phenol, provided by Reactivul (Romania), was used without further purification. Na₂PtCl₆·6H₂O (Fluka) was applied for the Pt/TiO₂ layers preparation. Demineralized water was used in all experiments.

Commercial fibreglass fabric (for technical laminate application, Skloplast, Slovak Republic) with specific weight of 500 g m⁻² and fibre diameter of 13 μm was used as a support material. The adhesive dress from the technical product was removed by firing at 400°C for 4 h.

The commercial Reinecke's salt from Aldrich was converted into the KCr(NH₃)₂(NCS)₄ and recrystallized, according to the procedure published by Wegner and Adamson [31].

2.2. Preparation of TiO₂ sol and Pt/TiO₂ supported on glass fibres

The TiO₂ sol corresponding to 5 wt.% of TiO₂ was synthesized by sol–gel technique as described in Ref. [32]. The glass fibres coated by Pt/TiO₂ were prepared by sol–gel technique analogously as in [30]. The concentration of platinum in the prepared Pt/TiO₂ photocatalysts was 1 mol.% Pt:Ti. The concentration of Pt/TiO₂ supported on the surface of glass fibres is 10 mg g⁻¹ of glass material.

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2.3. Apparatus and procedures

The photocatalytic phenol degradation was performed in the photoreactor constructed in our laboratory from glass that cuts off wavelengths below 300 nm and which transmittance at 310 nm is of 10% (Fig. 1). The standard emission lamps of identical size and electrical characteristics (power of 15 W) were applied as the irradiation sources. The spectral characteristics [33] for the model 3022 (labelled as Source I, maximum of intensity at 320 nm), model 3024 (Source II, maximum of intensity at 350 nm), model 3026 (Source III, maximum of intensity at 410 nm) and model 3028 (Source IV, white lamp resembling the solar spectrum) are shown in Fig. 2. The radiation flux of sources was determined applying Reinecke's salt actinometry [31,34,35]. This actinometric system allows the measurement of the light intensities in the wide range of wavelengths between 316 and 750 nm [31]. (Due to the transmittance of the reactor glass we can use Reinecke's salt actinometry also for Source I). The construction of the photoreactor enables the simple change of the irradiation source (Fig. 1). The prepared Pt/TiO₂ photocatalyst of 15 cm × 42 cm dimension was inserted into the photoreactor. The volume of irradiated phenol solution was 500 ml. The systems were continuously saturated by oxygen during irradiation. The oxygen flow was monitored using flowmeter. All photocatalytic experiments were carried out at the temperature of 50°C, actinometric experiments were performed at the temperature of 23°C.

The reflectance spectra of supported Pt/TiO₂ photocatalyst were measured using UV/visible spectrophotometer (M40, Zeiss, Germany) equipped by a reflectance accessory with an integration sphere.

The concentration of phenol and its main degradation product, hydroquinone, in the irradiated samples were determined by means of HPLC (FPLC Pharmacia, Sweden) using a Separon SIX C-18 column (Tessek, Czech Republic) and UV detector ($\lambda = 280$ nm). The mixture of methanol–water–H₃PO₄ (35:65:0.1) was applied as a mobile phase.

The UV spectra were measured using UV/visible spectrophotometer PU 8800 Philips.

3. Results and discussion

3.1. Blank experiments

The solutions with initial phenol concentration $c_0 = 50 \mu\text{mol dm}^{-3}$ were irradiated without Pt/TiO₂ photocatalysts at the temperature of 50°C with the oxygen flow of $2.7 \text{ dm}^3 \text{ ml}^{-1}$, utilizing all four light sources in the blank experiments. During 100 min of irradiation, no changes in the phenol concentration were found. Thus, the presence of Pt/TiO₂ photocatalyst is necessary for the phenol degradation by irradiation under the experimental conditions.

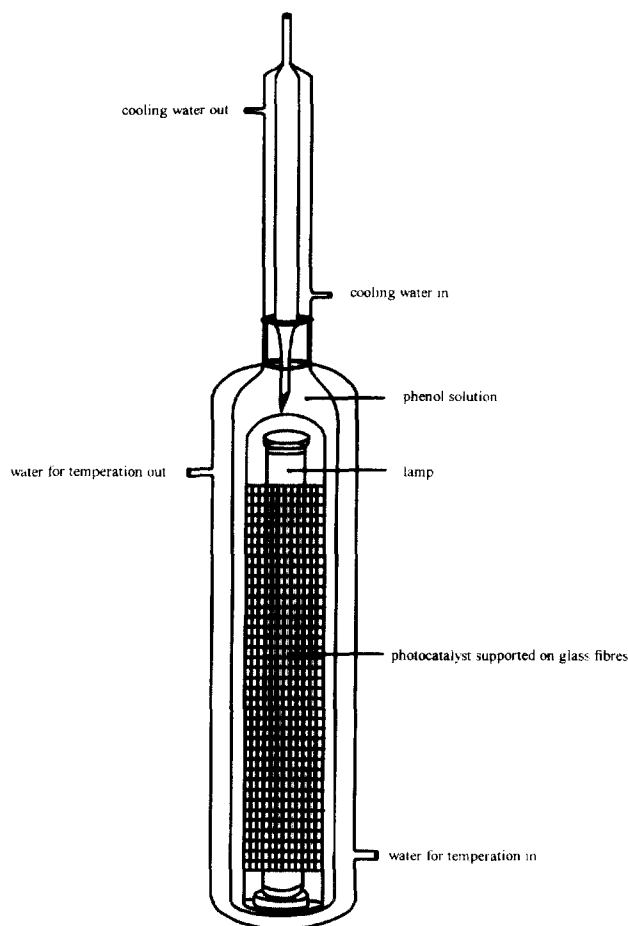


Fig. 1. The schematic illustration of the photoreactor used in our study.

3.2. Characterization of Pt/TiO₂ photocatalysts

The presence of platinum supported on the TiO₂ surface can significantly influence the photocatalytic activity of titanium dioxide [36–41]. Previously, we have studied the effect of platinum concentration on the phenol degradation rate, and the best properties were obtained for platinum content of 1.25 mol.% Pt:Ti. Concentration of Pt ≥ 5 mol.% Pt:Ti have detrimental effect on the photocatalyst activity [30], in accordance with the results published by Herrmann et al. [36], where the hyperbolic decrease of the reaction rate with the increased Pt loading on TiO₂ was interpreted by the electron transfer of photoelectrons from the semiconductor to metal particles, as well as by the decrease of the oxygen concentration photoadsorbed on TiO₂ as negatively charged species for increasing Pt content. According to Wang et al. [38] and Gerischer and Heller [39], the major role of small platinum clusters on TiO₂ is attributed to acceleration of superoxide radical anion O₂^{•-} formation and consequently with the effect of decreasing recombination. However, as the concentration of Pt on the surface increases the platinum deposits function as the recombination centres [40,41].

In this study, the photocatalyst Pt/TiO₂ (1 mol.% Pt:Ti) was supported on the glass fibres by sol–gel technique, the

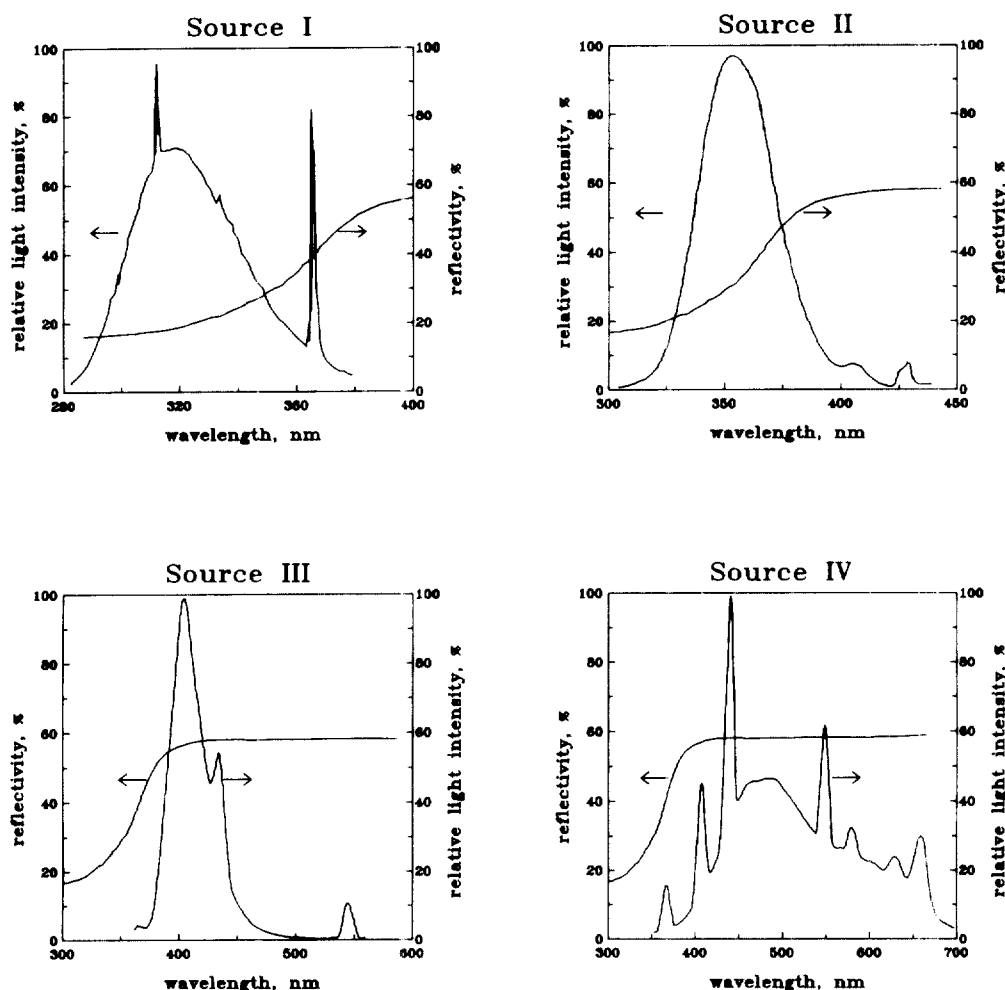


Fig. 2. Spectral characteristics of the illumination Sources I–IV along with the reflection spectrum of the prepared Pt/TiO₂ (1 mol.% Pt:Ti) photocatalyst supported on glass fibres.

concentration of photoactive layer on the glass textile was 10 mg g⁻¹. During the preparation procedure the material was heated at the temperature of 450°C for 4 h. The anatase structure is predominantly formed by this thermal treatment [30], and during preparation of the photocatalytic layer platinum was deposited on the TiO₂ surface, consequently the colour of glass fibres became metallic grey. According to the X-ray powder diffraction patterns previously measured for the Pt/TiO₂ material prepared by the same procedure, we confirmed the presence of Pt⁰ in the photocatalyst [30].

The reflectance spectrum of Pt/TiO₂ (1 mol.% Pt:Ti) supported on the glass fibres is depicted in Fig. 2 along with the spectral characteristics of the lamps used in our study.

The dependence of $(h\nu \cdot K)^{1/2}$ on the photon energy $h\nu$ (Fig. 3) is in the accordance with the theoretical equation [42–44],

$$h\nu \cdot \alpha = \text{const}(h\nu - E_{\text{bg}})^2 \quad (1)$$

where α is the absorption coefficient of Pt/TiO₂; E_{bg} is the energy band gap. (K is the Kubelka–Munk function calculated from the reflectance spectra [45] that is predetermined to be directly proportional to the absorption coefficient α).

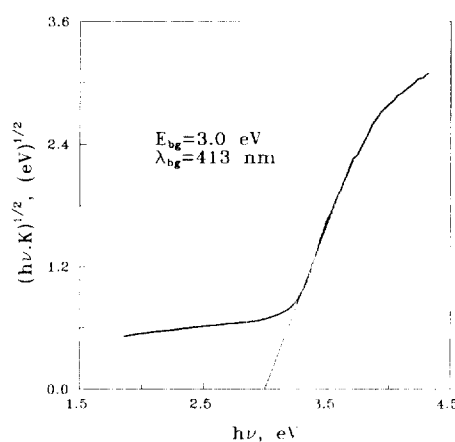


Fig. 3. The dependence of $(h\nu \cdot K)^{1/2}$ on the photon energy, $h\nu$, for the prepared Pt/TiO₂ (1 mol.% Pt:Ti) photocatalyst. The energy band gap E_{bg} was calculated by the extrapolation of the linear part ($\lambda_{\text{bg}} = 1240/E_{\text{bg}}$ [eV] [43]).

The value of energy band gap ($E_{\text{bg}} = 3.0$ eV, $\lambda_{\text{bg}} = 413$ nm) for photocatalyst Pt/TiO₂ (1 mol.% Pt:Ti) was determined by extrapolation of the linear part of the dependence as drawn in Fig. 3.

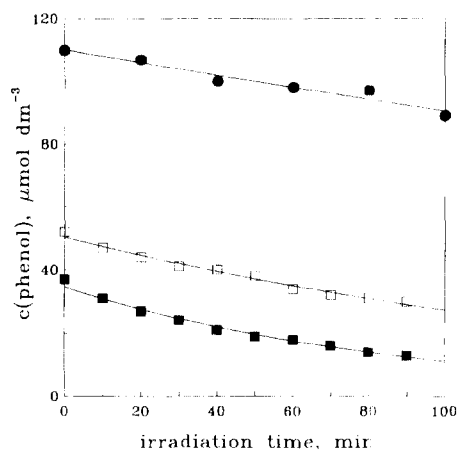


Fig. 4. The decrease in phenol concentration during irradiation using Pt/TiO₂ supported on glass fibres in systems with various initial phenol concentrations. Conditions: Source III; oxygen flow of 0.5 dm³ min⁻¹; temperature of 50°C.

3.3. Photocatalytic phenol degradation on Pt/TiO₂ supported on glass fibres

The decrease in phenol concentration during irradiation by the Source III (oxygen flow 0.5 dm³ min⁻¹) using Pt/TiO₂ supported on glass fibres in systems with various initial phenol concentrations is depicted in Fig. 4. The measured dependencies of phenol concentration, $c(\text{phenol})$, on the irradiation time, t_{irr} , were evaluated applying the formal first-order kinetics and the corresponding formal rate constants, k_f , and formal phenol half-life, $\tau_{1/2}$, were calculated using program SCIENTIST (MicroMath) according to the equations.

$$c(\text{phenol}) = c_0 \exp(-k_f t_{\text{irr}}) \quad (2)$$

$$\tau_{1/2} = \ln(2)/k_f \quad (3)$$

The linear dependence of the formal phenol half-life on the initial phenol concentration was found applying the Langmuir–Hinshelwood kinetic model for the TiO₂ photocatalysts immobilized on the glass fibres by calcination [29,30] or by sol–gel method [24,30,32], if the systems with constant oxygen concentration were irradiated. However, the deviations from the linearity were found for the Pt/TiO₂ photocatalysts used [30]. This effect was rationalized by considering the influence of platinum on the oxygen surface reactions as reported in Refs. [36–41].

Analogous non-linear dependence of the formal phenol half-life on the phenol initial concentration was observed in the presented photocatalytic experiments using Pt/TiO₂, and the half-life larger than 1200 min was determined for the irradiated systems characterized by initial phenol concentration of 1000 μmol dm⁻³. These experiments were performed with the main aim to find the optimal initial phenol concentration for its degradation under given experimental conditions, as the initial concentration of pollutants can sig-

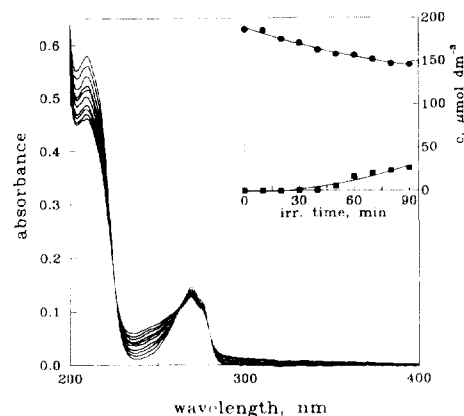


Fig. 5. A set of UV spectra measured during irradiation of phenol solution using Pt/TiO₂ supported on glass fibres. Inset represents the changes in the concentration of phenol (●) and hydroquinone (■) in the system. Conditions: Source III; $c_0 = 200 \mu\text{mol dm}^{-3}$; oxygen flow of 0.5 dm³ min⁻¹; temperature of 50°C.

nificantly influence the rate of their photocatalytic destruction [3,46]. According to the results obtained, we choose lower initial phenol concentration for the other sets of photocatalytic experiments in which the effect of oxygen flow and irradiation source has been evaluated.

The process of phenol degradation in the solution can be also easily monitored by UV spectroscopy as depicted in Fig. 5, where a set of UV spectra measured for variously irradiated samples is completed by the inset that illustrates the concentration profile of phenol and hydroquinone during irradiation. Owing to the relatively low conversion of phenol the analysis of the second main intermediate, i.e., catechol [47], was not possible.

3.4. Effect of the oxygen flow on the phenol degradation using Pt/TiO₂ supported on glass fibres

The presence of oxygen in the photocatalytic systems can significantly affect the fate of the photogenerated species, i.e., it reacts with the conduction band electrons to form superoxide anion radical, and it combines with organic radicals generated by the hydroxyl radical attack or by the reaction with photoholes [38–40].

Additionally, in our study oxygen has a further important role. The oxygen flow serves as the stirring medium performing the mass transfer in the irradiated systems. Fig. 6 illustrates the linear decrease of the formal phenol half-life on the increased oxygen flow in the photocatalytic degradation on Pt/TiO₂ upon irradiation by Source III ($c_0 = 50 \mu\text{mol dm}^{-3}$). The increasing oxygen flow significantly enhances the effectiveness of photocatalytic process confirming the mass transfer limitations in accordance with other studies using immobilized TiO₂ photocatalysts [29,48–51]. However, the oxygen flow larger than 5 dm³ min⁻¹ under given experimental conditions damaged the photocatalyst's quality.

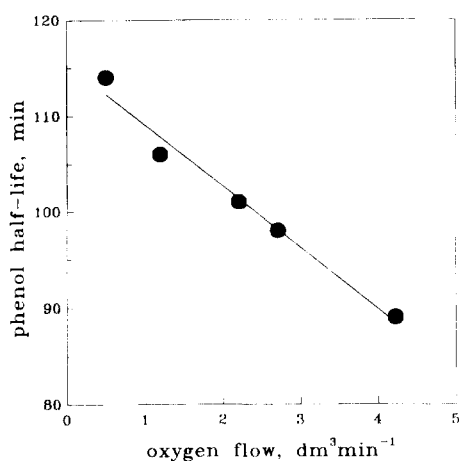


Fig. 6. Dependence of the formal phenol half-life on the oxygen flow for the photocatalytic reactions using Pt/TiO₂ photocatalysts supported on glass fibres. Conditions: Source III; $c_0 = 50 \mu\text{mol dm}^{-3}$; temperature of 50°C.

3.5. Effect of the light source on the phenol degradation using Pt/TiO₂ supported on glass fibres

The effect of the light source utilized on the phenol photocatalytic degradation efficiency was evaluated by means of the apparent quantum yield, q_{app} , which was calculated using the following equation

$$q_{\text{app}} = \frac{\Delta n_{\text{phenol}}}{t \Phi_{\text{abs}}} = \frac{c_0 V}{2 \tau_{1/2} \Phi_{\text{abs}}} \quad (4)$$

where c_0 is the initial concentration of phenol [mol dm^{-3}]; V is the irradiated volume [dm^3]; $\tau_{1/2}$ is the formal phenol half-life [s]; Φ_{abs} is the light flux absorbed by the Pt/TiO₂ photocatalyst [mol s^{-1}].

The incident photon flux of the individual sources was determined by Reinecke's salt actinometry [31,34,35]. The actinometric experiments were performed under the strictly identical experimental conditions with the photocatalytic experiments.

For the q_{app} calculation we have used the following approximations [52]: (i) the average quantum yield of Reinecke's salt actinometer calculated as the arithmetic mean of the quantum yields at the wavelengths emitted that are actual for the individual Source I–IV. (The values of Φ_{em} were calculated according to the procedure described in Refs. [31,34,35]); (ii) wavelength selective correction for Pt/TiO₂ (1 mol.% Pt:Ti) absorbance were involved in the calculation of Φ_{abs} ; (iii) the actinometric experiments were arranged to reach the complex absorption of the incident flux in the reaction system.

The calculated apparent quantum yields of phenol degradation along with the formal phenol half-life and calculated radiation fluxes for the photocatalytic phenol degradation on Pt/TiO₂ ($c_0 = 100 \mu\text{mol dm}^{-3}$, oxygen flow of $2.7 \text{ dm}^3 \text{ min}^{-1}$) are summarized in Table 1. The values of q_{app} are in a good agreement with that reported for the photocatalytic degradation of chlorosalicylic acid using immobilized TiO₂ catalysts synthesized by sol-gel technique [24].

The largest value of q_{app} was obtained for the Source I with maximum of intensity at 320 nm in accordance with results published by Stafford et al. [53], where the quantum yield of photocatalytic 4-chlorophenol removal significantly increased with decreasing wavelength. This effect was explained in terms of the higher energy electrons being less likely to recombine than electrons excited into the lowest unoccupied molecular orbital [53].

We consider, additionally, the effect of light intensity on the apparent quantum yield (Fig. 7), and we measured very good correlation for the linear dependence $q_{\text{app}} \sim \Phi^{0.5}$ ($R^2 = 0.998$). For the heterogeneous photocatalytic reactions the non-linear behaviour of the reaction rate on the light intensity was observed [46], and the apparent quantum yield of photocatalytic reaction is proportional to $\Phi^{0.5-0.74}$ [53,54]. The effect of light intensity was explained in terms that at increasing electron-hole generation rate determined by the high radiation intensities the rate of electron-hole recombination predominates with respect to the rate of electron-hole capture by species involved in the chemical reactions [54].

Despite the lower efficiency, the photocatalytic degradation of phenol using Pt/TiO₂ photocatalysts supported on glass fibres is also possible by the light of Source IV resem-

Table 1

The values of formal phenol half-life and the apparent quantum yield evaluated for the phenol decomposition using Pt/TiO₂ photocatalysts supported on glass fibres along with the values of radiation fluxes used in the quantum yield calculation

Source	$\tau_{1/2}$ [min]	Φ_{em} [mol s^{-1}]	Φ_{abs} [mol s^{-1}]	q_{app}
I (320 nm)	240	6.8×10^{-7}	2.0×10^{-7}	0.0085
II (350 nm)	182	7.0×10^{-7}	3.0×10^{-7}	0.0075
III (410 nm)	98	3.2×10^{-6}	1.5×10^{-6}	0.0029
IV (white lamp)	289	5.5×10^{-6}	2.1×10^{-6}	0.0007

$c_0 = 100 \mu\text{mol dm}^{-3}$,
Oxygen flow of $2.7 \text{ dm}^3 \text{ min}^{-1}$,
Temperature of 50°C.

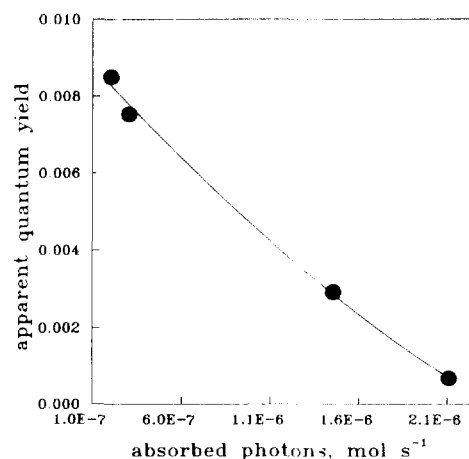


Fig. 7. Dependence of the apparent quantum yield of phenol degradation on the light intensity of the Sources I–IV.

bling the solar spectrum, so the prepared photocatalysts may be used also for solar applications.

4. Conclusions

The photocatalytic degradation of phenol on Pt/TiO₂ (1 mol.% Pt:Ti) supported by sol-gel technique on glass fibres was measured when four different 15 W standard emission lamps were used as irradiation sources. The illumination source applied significantly influences the value of apparent quantum yield of phenol degradation. The best results were obtained for the emission lamps with spectral distribution below 400 nm (Source I and II). According to the results obtained using light Source IV resembling the solar spectrum, the prepared Pt/TiO₂ photocatalysts are applicable also for solar applications.

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