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Development of new dyeing photoinitiators based on 6*H*-indolo[2,3-*b*]quinoxaline skeleton

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Abstract

The series of new dyes, which structures are based on 6*H*-indolo[2,3-*b*]quinoxaline skeleton that possess characteristic electronic absorption band at a boundary of UV and visible light were tested as potential light absorbing chromophores for photoinitiated polymerization.

The studied dyes can be classified into two different groups. The first is the group, so called 'the branched dyes', which structures possess the part of molecule that can rotate without restraints and are characterized by low photoinitiation ability. The second, planar and rigid group of molecules provides another chromophores, which possess quite different properties in comparison to that observed for the branched dyes. Their photoinitiation ability is comparable to that observed for many commercially available photoinitiating systems.

The location of electronic absorption spectra at a boundary of UV and visible light makes the tested dyes the good candidates for the photoinitiating system applied in dental restorative materials. Their high molar absorption coefficient allows to decrease the dyes concentration in dental formulation in comparison to commonly used camphorquinone.

Keywords: Photopolymerization; Dyeing photoinitiators; 6*H*-Indolo[2,3-*b*]quinoxalines

1. Introduction

The process of photoinitiated polymerization may be started by direct photolysis of a precursor providing free radicals by bond dissociation. The radicals may also be formed in bimolecular processes. Panchromatic sensitization of photoinitiated polymerization often requires the presence of suitable dye as a light absorber. This can either transfer energy, or undergo electron transfer process, which uses a light to initiate electron transfer from a donor to an acceptor molecule. The process is possible because electronically excited states are both better oxidants and better reductants than their ground states equivalents. Translating these to the sensitization of free radical polymerization, one can anticipate that two types of sensitization via electron transfer process should occur.

(1) Dye photoreduction leading to subsequent polymerization was first reported in 1954 [1] when Oster identified several groups of dyes that are photoreduced in the presence of suitable reductants like ascorbic acid and triethanolamine. The dyes included the acridines, xanthenes and thiazines.
(2) Dye photooxidation leading to subsequent polymerization requires molecules that are strong electron acceptors in the ground state. Systems comprised of dyes and *N*-alkoxyheterocycles acting as one electron acceptors are examples of such systems and photoinitiators for the polymerization of acrylates [2,3].

Marcus [4–6] provided a simple approach allowing the prediction of the kinetics of the electron transfer process, using thermodynamic and spectroscopic parameters characterizing both an electron donor and an electron acceptor. Depending on the thermodynamics of the electron exchange, it is possible to quantitatively predict a relationship between the free energy of activation for electron transfer and the free energy associated with photoinduced electron transfer process. Beside the thermodynamics of the electron transfer process, one should take into account the facts that the photoinitiation ability of the

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photoredox pair depends on the nature of the reacting partners that may generate either radical ions or neutral radicals that undergo fragmentation reactions yielding radicals and ions [7–9].

Very often, however, the research devoted to colored photoinitiators is determined by a potential application, particularly in the area of imaging science [10], steroelithography [11], coating technology [12] or in dentistry [13].

In this paper, it is our intention to present the studies on the series of new dyes, which structures are based on 6*H*-indolo[2,3-*b*]quinoxaline skeleton that possess characteristic electronic absorption band at a boundary of UV and visible light. The location of electronic absorption spectra in this region makes the tested dyes the potential photoinitiating systems that can be initiated by the commercial dental lamps, e.g. can be applied for preparation of dental restorative materials. Their high molar absorption coefficient allows to decrease its concentration in dental formulation in comparison to commonly used camphorquinone [14].

2. Experimental

Substrates used for the preparation of the dyes and electron donors were purchased from Fluka, Merck or Aldrich. Monomer, 2-ethyl-2-(hydroxymethyl)-1,3-propanediol triacrylate (TMPTA), 1-methyl-2-pyrrolidinone (MP), *N*-phenylglycine (NPG), thiophenoxyacetic acid (TPAA), phenoxyacetic acid (PAA), ethyl 4-dimethylaminobenzoate (EDAB), camphorquinone (CQ), were purchased from Aldrich. *N*-(4-cyano-phenyl)glycine (CN-NPG) used as electron donor was prepared according to procedure given by Dyer et al. [15].

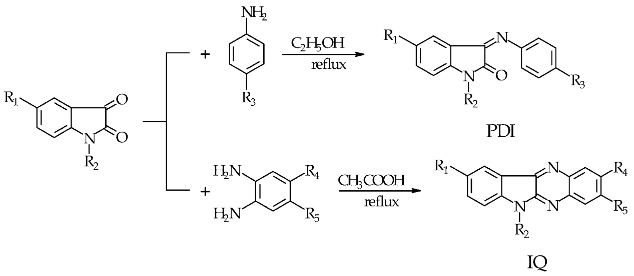
Absorption spectra were obtained using a Varian Cary 3E spectrophotometer. Fluorescence spectra were recorded using a Hitachi F-4500 spectrofluorimeter. Absorption spectra were recorded for ethyl acetate solutions. Both fluorescence and phosphorescence spectra were recorded for 2-methyltetrahydrofuran solutions. Fluorescence quantum yield was measured using 9-methylantracene as the actinimeter [16]. The nanosecond laser flash photolysis experiments were performed using a LKS.60 Laser Flash Photolysis apparatus (Applied Photophysics). Laser irradiation at 355 nm from the third harmonic of the Q-switched Nd:YAG laser from a Lambda Physik/model LPY 150 operating at 65 mJ/pulse (pulse width about 4–5 ns) was used for the excitation.

The reduction and oxidation potentials of the dyes were measured by cyclic voltammetry. An Electroanalytical MTM (Krakow) Model EA9C-4z was used for measurements, and Ag–AgCl electrode served as a reference electrode. The supporting electrolyte was 0.5 M tetrabutylammonium perchlorate.

The kinetics of free radical polymerization were studied using a polymerization solution composed of 1 ml of MP and 9 ml of TMPTA. Dye concentration was varied from 1.5×10^{-3} to 2.5×10^{-3} M; concentrations of electron donor were 0.1 M. The polymerizing mixture was not deareated.

TMPTA-MP kinetics of polymerization measurements were carried out by measuring the polymerization heat evolution of a sample, irradiated with a dental lamp (Luxor Model 4000) and an Innova 90-4 argon-ion laser (351 and 361 nm lines) through an optical system, in a home-made micro-calorimeter as described earlier [17–19]. The irradiation intensity measured with a Coherent Power Meter Type 543–500 mA and was 25.5 mW/cm^2.

Synthesis. For the study, two different groups of the dyes were synthesized by the condensation reaction (Scheme 1) of corresponding aniline or 1,2-phenylenediamine with isatin (indole-2,3-dione) that yields in the first case an appropriate 3-phenylimino-1,3-dihydro-indol-2-one [20,21] (PDI, branched dyes) and in the second case an appropriate 6*H*-indolo[2,3-*b*]quinoxaline [22–24] (IQ, planar dyes).



Scheme 1.

where: $R_1 = H, Br, NO_2$; $R_2 = H, CH_3, C_6H_5$; $R_3 = H, I$; $R_4 = R_5 = H, Cl$.

The final products were purified with the use of preparative thin-layer chromatography. The compounds were characterized by proton and carbon NMR spectroscopy using the Varian Gemini 200 spectrometer.

3. Results and discussion

The structures summarized in Table 1 have been investigated. The compounds PDI-H, PDI-CH3, PDI-I and PDI-Br possess the part of molecule (phenyl azomethine residue) that can rotate without restraints (branched dyes).

The shapes of the absorption spectra of all branched dyes in ethyl acetate are shown in Fig. 1. The absorption spectra show two bands at approximately 300 and 415 nm. This group of the dyes emits very weak fluorescence with the maximum located at approximately 460 nm. Table 1 shows the absorption and fluorescence data for the studied branched dyes. The change of the dye structures leading to the formation of planar and rigid molecules provide

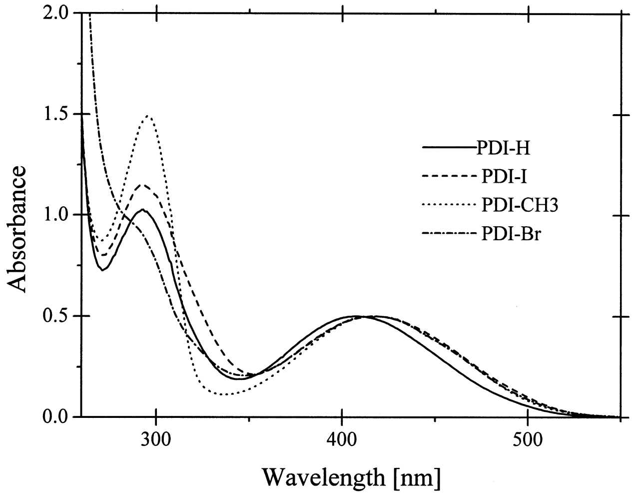


Fig. 1. The electronic absorption spectra of the branched dyes obtained in ethyl acetate solution.

another chromophores, which possess quite different properties in comparison to that observed for branched dyes. A typical example of the spectroscopic properties in these chromophores (IQCH3) is shown in Fig. 2. It should be noted that in ethyl acetate the type of substituent introduced into dye molecule has a minor influence on the shape of the spectrum in general case.

Table 1 summarizes also all spectroscopic properties determined for this class of the dyes. The most noticeable features for comparison in Figs. 1 and 2 and Table 1 are the changes in the shape and the position of the electronic absorption spectra, significant enhancement of the fluorescence quantum yield and, finally, the presence of phophorescence spectra, which indicates the formation of the triplet state. The triplet state energy, as is summarized in Table 1, ranges from 224 to 258 kJ mol^{-1}. The formation of the triplet state can be additionally confirmed by the laser flash photolysis experiments. Fig. 3 depicts the time resolved triplet–triplet transient spectra obtained for IQCH3 in MeCN solution. The analysis of the decay of the transient absorption at 370 nm suggests the presence of one component with the lifetime of about 1.45 μs. The spectrum is not observed in oxygen saturated solution and this allows to attribute its occurrence to the presence of the triplet state.

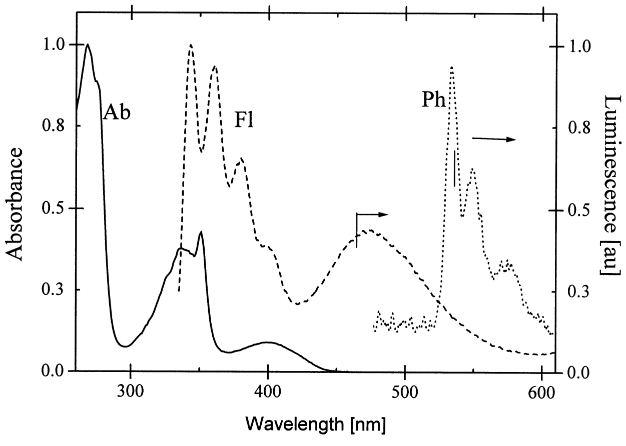


Fig. 2. The spectroscopic properties of IQCH3. The absorption spectra recorded in ethyl acetate, the emission spectra recorded in 2-methyltetrahydrofuran.

The electrode potentials at which studied dyes undergo reduction were determined by cyclic voltammetry. The cyclic voltammograms for the dyes under investigation show one electron reversible reduction. The values of the reduction potentials for the dyes tested are also summarized in Table 1.

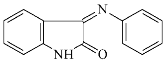
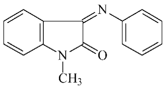
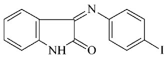
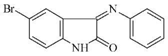
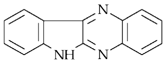
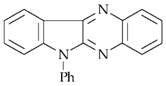
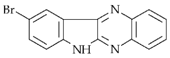
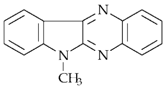
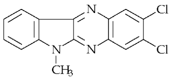
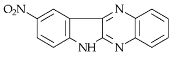
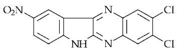
The measured electrochemical and spectroscopic parameters allow the calculation of the free energy activation (ΔG_{el}) for the photoinduced electron transfer process. The calculated, using the Rehm–Weller equation, [25,26] thermodynamical properties of dye-*N*-phenylglycine derivative photoredox pair are listed in Table 1. For the calculation of ΔG_{el} for branched dyes the energy of the 0,0 transition for the singlet excited state was taken into account and for planar dyes the energy of the 0,0 transition for the triplet state, respectively. It is well known from the basic Rehm–Weller requirement that the free energy of activation for the photoinduced electron transfer (ΔG_{el}) process should have a negative value. As it can be seen in Table 1 the free energy change for the electron transfer for entire series of the tested dyes is negative. It means that the photoinduced electron transfer for the dye-*N*-phenylglycine derivative bimolecular system is thermodynamically allowed.

The absorption of the dyes pictured in Figs. 1 and 2 reveal that both groups of tested dyes possess broad spectra with a long-wavelength part located in the blue region of the visible light. Additionally the planar dyes (IQ) absorb (see Table 1) in the near UV (300–400 nm) of the optical radiation spectrum. These features make tested dyes the good candidates for the photoinitiating system applied in dental restorative materials and UV cured formulations as well. For this reason the polymerization photoinitiation ability of the branched and planar dyes were tested with the use of an argon-ion laser emitting in near UV region (351 and 361 nm) and with the use of a dental lamp.

The efficiency of photoinitiation of free radical polymerization of tested dyes depends strongly on the dye structure. Fig. 4 presents the photoinitiated polymerization kinetic traces recorded for PDI-CH3 and IQCH3.

It is apparent from the inspection of the experimental data shown in Fig. 4 that the precursor, branched dye, initiates polymerization much less efficiently in comparison to the its planar analogue. In our earlier papers we have demonstrated that the elimination or limitation of the rotation of the phenyl group in the azomethine part of molecule causes a significant increase of the photoinitiation ability observed for other, different type of azomethine dyes [27–29]. Elimination of the motion of C=N bond by

Table 1
Structures and basic spectroscopic, thermodynamical and electrochemical properties of investigated dyes

Dye	Mark	λ_{max} [a] (nm)	ε ($dm^3\ mol^{-1}\ cm^{-1}$)	λ_{max}^{Fl} [b] (nm)	ϕ_{Fl}	E_T^{00} (kJ/mol)	E_S^{00} (kJ/mol)	E_{red} (V)	ΔG^0 (eV)[c]	ΔG^S (eV)[d]
	PDI-H [20]	408	2400	460	1.1×10^{-4}	–	276	−0.79	–	−1.64
	PDI-CH3	415	1100	459	1.6×10^{-4}	–	263	−0.94	–	−1.37
	PDI-I [21]	420	3000	459	1.9×10^{-4}	–	264	−0.98	–	−1.33
	PDI-Br	418	2700	458	1.9×10^{-4}	–	260	−0.96	–	−1.31
	IQH [22]	334 350 386	14100 16300 4200	465	0.039	227	–	−1.17	−0.48	–
	IQPh	335 351 396	16400 19500 4100	475	0.035	227	–	−1.21	−0.44	–
	IQBr [23]	335 352 390	20100 25000 5400	468	0.023	258	–	−1.16	−0.81	–
	IQCH3 [24]	334 351 405	14100 16500 3400	476	0.040	226	–	−1.18	−0.45	–
	IQCH3Cl2	344 361 409	19700 26700 4800	475	0.053	256	–	−1.18	−0.78	–
	IQNO2 [23]	304 364 399	10000 12600 10500	473	0.010	230	–	−1.18	−0.50	–
	IQNO2Cl2	309 355 409	12000 14200 11000	468	0.016	224	–	−1.20	−0.41	–

[a] Ethyl acetate.

[b] 2-Methyltetrahydrofuran.

[c] Measured for *N*-(4-cyano-phenyl)glycine (CN-NPG) as electron donor ($E_{ox} = 0.707$ V) [18,19].

[d] Measured for *N*-phenylglycine (NPG) as electron donor ($E_{ox} = 0.426$ V) [18,19].

co-planarization of the azomethine residue with other parts of the dye decreases the degree of branching of the dye, and this stabilizes the molecule in its excited state. This distinct effect is easy to interpret in terms of the activation energies needed for aromatic imine isomerization, which is in order of 20 kcal/mol or less. One more important conclusion arises from the analysis presented; that is, the rotation around C=N bound is the main channel of the dye excited state deactivation [30].

It is worth noting that the photoinitiation capability for the group of the planar dyes also depends on the dye structure. This is illustrated in Fig. 5.

The comment requires low photoinitiation capabilities demonstrated by IQNO2 and IQNO2Cl2. From literature it is known that nitro compounds in the presence of electron donors undergo photoreduction yielding corresponding nitroso derivative [31], which are known to be efficient free radical scavengers [32]. It is also noteworthy fact that nitro compounds themselves are considered as efficient inhibitors of free radical polymerization [33]. The above mentioned properties seem to explain an unexpected lowering in photoinitiation ability of IQNO2 and IQNO2Cl2.

The efficiency of photoinitiated polymerization, for the entire family of studied dyes, depends not only on the type of a dye, but is strongly affected by a type of an electron donor. This is shown in Fig. 6.

The data characterizing the photoinitiation capability of tested photoredox pairs exhibit a wide range of values (summarized in Table 2) that depend on the nature of both, a dye and an electron donor. On the basis of the data listed in Table 2 one can conclude that there is a quite significant

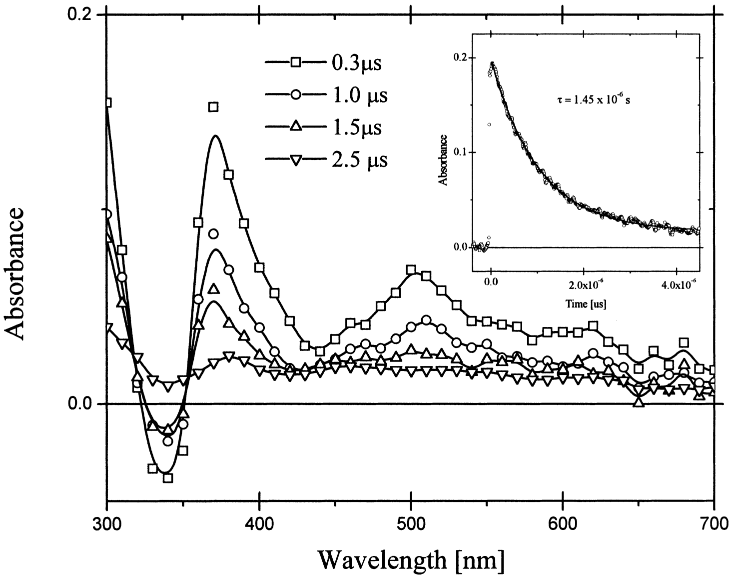


Fig. 3. Time resolved transient absorption spectra of IQCH3 in argon saturated acetonitryle. Inset shows the experimental trace for the decay of the triplet state.

heavy atom effect on the photoinitiation capability of the tested dyes. It should be noted that in Table 2 the data characterizing the photopolymerization ability for two, the best and the worst electron donors tested are listed. The use of thiophenoxyacetic acid, ethyl 4-dimethylaminobenzoate and *N*-phenylglycine yield the formulations of lower sensitivity in comparison to the dyes-CN-NPG photoredox pairs.

We showed earlier that the rate of photoinitiated polymerization can be expressed by the equation [17,18].

$$R_p = -\frac{d[M]}{dt} = k_p[M]\sqrt{\frac{I_a k_{el}}{k_t}} \qquad (1)$$

The Eq. (1) predicts that the rate of the polymerization might be in part controlled by the rate of photoinduced electron transfer. The experimental verifications of the Eq. (1) for the tested photoinitiating systems is possible by measuring the rate of the primary process of the polymerization initiation process, e.g. the rate of the electron transfer between the tested dyes and the electron donors and the comparison of these rates to the rates of photoinitiated polymerization.

The quenching rate constants, k_q (since the quenching of the dyes triplet by the electron donor is the only way of triplet deactivation, therefore under this condition one can assume that $k_q = k_{el}$), were obtained from the experimentally measured pseudo-first-order rate constant, k_{obs}, for the

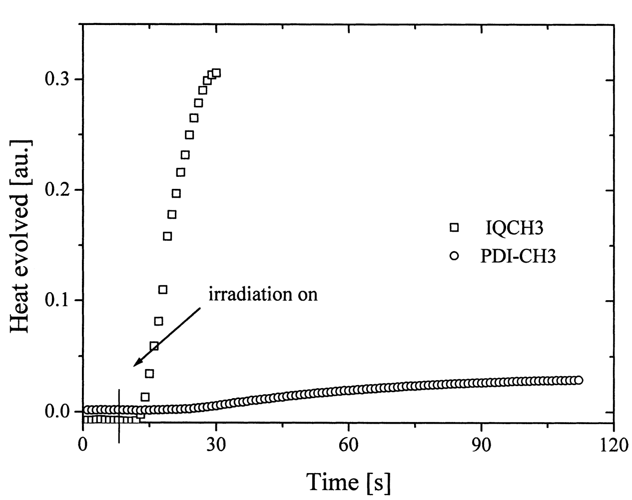


Fig. 4. Photopolymerization kinetic traces recorded for an initial time of polymerization of TMPTA-MP (9:1) monomer formulation. Initiating system: dye—marked in the figure; electron donor *N*-phenylgycine (NPG). Light intensity of an argon-ion laser irradiation 25.5 mW/cm^2.

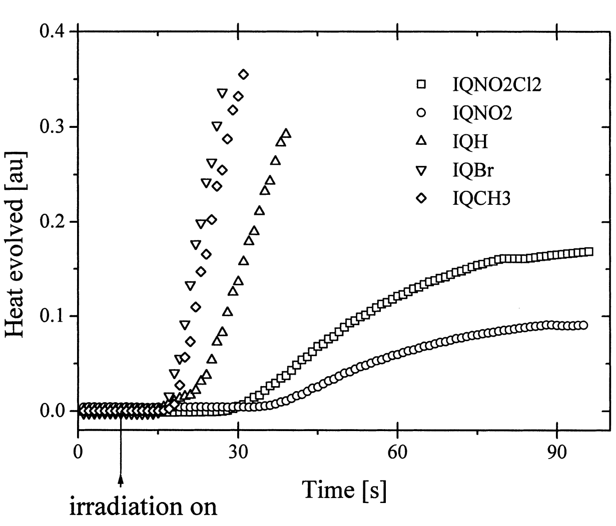


Fig. 5. The family of the photopolymerization kinetic traces recorded for novel tested dyes (marked in Figure). Formulation: TMPTA-MP (9:1), electron donor: *N*-(4-cyano-phenyl)glycine (0.1 M). Light source dental lamp—irradiation intensity equal 25.5 mW/cm^2.

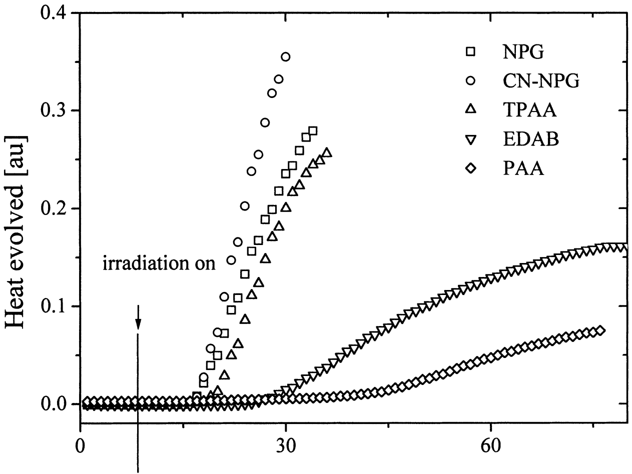


Fig. 6. Photopolymerization kinetic traces recorded for an initial time of polymerization TMPTA-MP (9:1) monomer formulation. Initiating system: dye—IQCH3 and various electron donors (0.1 M) (marked in the legend, where: NPG-*N*-phenylglycine, CN-NPG—*N*-(4-cyano-phenyl)glycine, TPAA—thiophenoxyacetic acid, EDAB—ethyl 4-dimethylaminobenzoate and PAA—phenoxyacetic acid). Light source dental lamp—irradiation intensity equal 25.5 mW/cm^2.

decay of the dyes triplet by the formula

$$k_{\mathrm{obs}} = \tau_T^{-1} + k_{\mathrm{q}}[\mathrm{ED}] \tag{2}$$

where τ_T is the lifetime of the dye triplet in the absence of an electron donor (ED). Some typical plots based on Eq. (2) are presented in Fig. 7 and the quenching rates constants obtained for *N*-(4-cyano-phenyl)glycine as electron donor are summarized in Table 2.

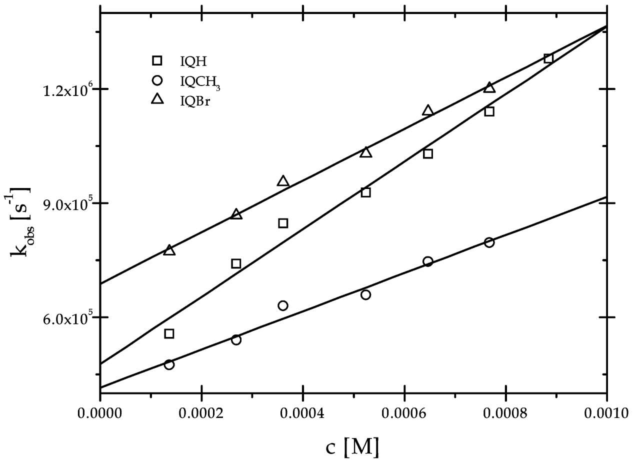


Fig. 7. Plots according to Eq. (2) for tested dyes triplet quenching. As electron donor *N*-(4-cyano-phenyl)glycine was applied.

Fig. 8 presents the relationship between the rates of photoinitiated polymerization and the rate constants of the electron transfer process.

It is apparent from the inspection of the data shown in Fig. 8 that there is no linear correlation (as predicted by Eq. (1)) between the rates of photoinitiated polymerization and the rate constants of the electron transfer process. The comparison of the Marcus plot (see inset in Fig. 8) obtained for tested dyes and relationship between the rate of polymerization versus the rate constant of triplet state quenching, indicates, that there is another factor governing the kinetics of the photoinitiated polymerization. The measured rate constants of the electron transfer process (close to the diffusional limit) and observed Marcus

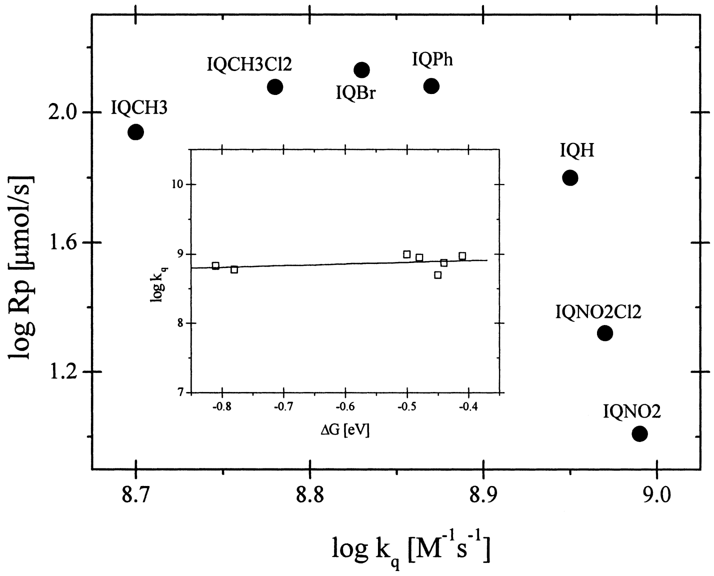


Fig. 8. Correlation between the rates of the electron transfer process and the rates of photoinitiated polymerization. Inset: the Marcus plot for the studied IQ dyes triplet state quenching by *N*-(4-cyano-phenyl)glycine.

Table 2
The photophysical properties and measured rates of photoinitiated polymerization for studied dyes in the presence of selected electron donors

Dye	$\tau_T \times 10^6$ (s)	*N*-(4-Cyano-phenyl)glycine			Phenoxyacetic acid	
		k_q ($\times 10^8$ M^{-1} s^{-1})	R_p (μmol/s) (laser)	R_p (μmol/s) (lamp)	R_p (μmol/s) (laser)	R_p (μmol/s) (lamp)
IQBr	2.08	6.78	134.51	78.12	46.19	14.74
IQPh	1.82	7.45	121.24	74.17	19.05	10.49
IQCH3Cl2	1.74	5.99	119.80	71.62	10.37	7.88
IQCH3	1.45	5.01	88.22	66.18	9.66	5.29
IQH	1.31	8.88	63.56	40.59	3.78	2.70
IQNO2Cl2	1.18	9.35	21.14	10.23	No polymer	No polymer
IQNO2	0.38	9.90	11.94	5.67	No polymer	No polymer

Laser irradiation intensity equal 25.5 mW/cm^2, dental lamp irradiation intensity equal 25.5 mW/cm^2.

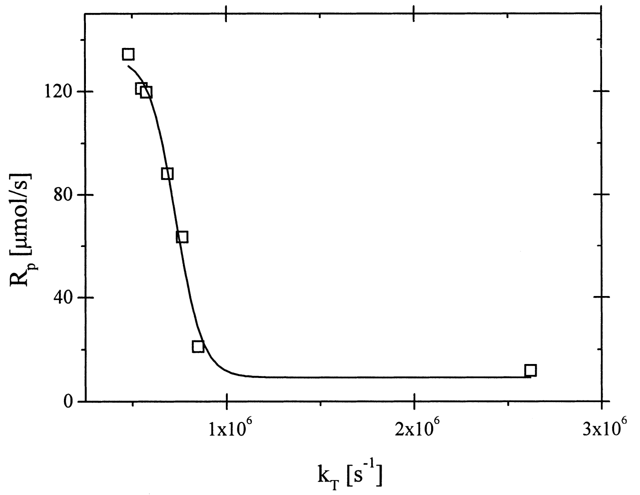


Fig. 9. Relationship between the rates of photoinitiated polymerization and the rate constants of triplet state decay for tested IQ dyes.

relationship suggest that an overall efficiency of photoinitiated polymerization might be controlled by the diffusion.

Verification of this hypothesis may come from the comparison of the triplet state decay rate constants (measured using laser flash photolysis method) and the polymerization rates. Fig. 9 shows this type of relationship.

The factor needed for the explanation of the observed phenomena is the viscosity of the monomers formulation used for the photoinitiated polymerization experiments. The measurements performed for TMPTA-MP (9:1) mixture gives viscosity value equal $\eta = 37.7$ m Pa s. This value corresponds to the time independent bimolecular rate constant reaction determined based on Smoluchowski's theory to be $k_{\mathrm{diff}} = 1.61 \times 10^5$ l mol^{-1} s^{-1}. The calculated rate constant clearly indicates that the polymerization initiated by studied dyes occurs in the transition region of diffusion and non-diffusion-controlled reactions.

As it was mentioned earlier, the absorption spectra of IQ dyes make them the good candidates for the photoinitiating

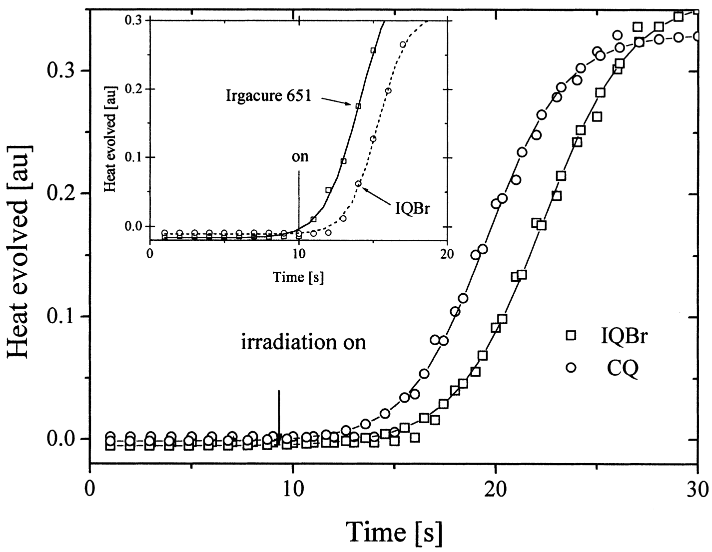


Fig. 10. Comparison of the kinetics of photoinitiated polymerization initiated by IQBr-CN-NPG and CQ-CN-NPG photoredox pair. Light source dental lamp—irradiation intensity equal 25.5 mW/cm^2. Inset: polymerization initiated by IQBr-CN-NPG and Irgacure 651 systems. Light intensity of an argon-ion laser irradiation 25.5 mW/cm^2.

systems applied in dental restorative materials. The above conclusion can be supported by the comparison of the kinetics of polyolacrylate polymerization initiated by IQBr-CN-NPG photoredox pair and camphorquinone, a commonly used in dentistry photoinitiator [14].

As the data presented in Fig. 10 show, IQBr-CN-NPG demonstrates identical in comparison to CQ-CN-NPG initiating photoredox pair photoinitiation ability. Similar conclusion can be derived from the comparison of photoinitiation sensitivity observed for IQBr-CN-NPG and Irgacure 651 (Fig. 10 inset).

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