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Photophysical Properties and Electrochemistry of the N,N′-bis-n-butyl Derivative of Naphthalene Diimide

Yevgen POSOKHOV[1,2], **Serap ALP**[3], **Banu KÖZ**[1], **Yusuf DİLGİN**[4], **Sıddık İÇLİ**[1*]
[1] *Solar Energy Institute, Ege University, Bornova, 35100 İzmir-TURKEY*
[2] *Institute for Chemistry at Kharkov National University,61077 Kharkov-UKRAINE*
[3] *Department of Chemistry,Faculty of Science and Arts, Dokuz Eylül University, Buca, İzmir-TURKEY*
[4] *Department of Chemistry, Ege University, Bornova, 35100 İzmir-TURKEY*
e-mail: icli@bornova.ege.edu.tr

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The spectral-luminescent properties and electrochemistry of the N,N′-bis-n-butyl derivative of naphthalene diimide were studied. The main photophysical parameters (quantum yield, rate constants of fluorescence and of non-radiative deactivation and fluorescence lifetime) were determined.

The LUMO value (–3.51 eV) was determined by cyclic voltammetry. The N,N′-bis-n-butyl derivative of naphthalene diimide (BBND) shows 2 reversible reduction steps (-1.19 V and –1.75 V vs. Ferrocene) in chloroform.

Quenching the fluorescence emissions of aromatic donor molecules in acetonitrile revealed the electron acceptor ability of the studied naphthalene diimide derivative.

It has been found that, in contrast to perylene diimide derivatives, which form π-electron donor-acceptor complexes with aromatic donor molecules of naphthalene, phenanthrene, pyrene and perylene, BBND does not form π-electron-donor acceptor complexes with aromatic donor molecules.

Key Words: Spectral-luminescent properties, cyclic voltammetry, N,N′-bis-n-butyl derivative of naphthalene diimide.

Introduction

There has been increasing interest in 1,4,5,8-naphthalene-diimide derivatives in recent years, mainly due to their electron acceptor properties[1,2], which make them suitable for a series of applications, including the formation of Langmuir-Blodgett films[3], the preparation of electrically conducting materials[4,5], π-stacked materials absorbing in the near-IR region[6] and nanotube-like structures[7] and as models for the photosynthetic reaction center[8–10].

In this report, we present the spectral-luminescent, photophysical and electrochemical characterization of N,N′-bis-n-butyl derivative of naphthalene diimide.

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[*] Corresponding author

Experimental

Materials and methods

Chemicals

The N,N′-bis-n-butyl derivative of naphthalene diimide was prepared by the method employed for the synthesis of N-substituted perylene diimides[11]. 1,4,5,8-Naphthalene-dianhydride was reacted with an appropriate amine derivative in *m*-cresol/isoquinoline at elevated temperatures (>200 °C) for 4-6 h in order to prepare the naphthalenediimide. Molecular structures were analyzed by means of IR and ^{1}H NMR.

1,4,5,8-Naphthalenedianhydride, n-butylamine, naphthalene, phenanthrene, pyrene, perylene and acetonitrile were obtained from Fluka and Merck, and received as they are.

Synthesis of N,N′-bis-n-butyl-l,4,5,8-naphthalenediimide

A mixture of 1,4,5,8-naphthalenedianhydride (1 g, 3.75 mmol) and n-butylamine (0.7 g, 10 mmol) was dissolved in 20 mL of *m*-cresol and a few drops of isoquinoline were added. The temperature was gradually increased to 160 °C. The mixture was kept at this temperature for 6 h under nitrogen. The viscous solution was diluted with 20 ml *m*-cresol and poured slowly into 50 mL of methanol while stirring. The precipitate was filtered and washed thoroughly with warm acetone. Crude product was purified by column chromatography, using dichloromethane as the eluent. N,N′-bis-n-butyl-l,4,5,8-naphthalenediimide, $C_{22}H_{24}N_2O_4$, MW: 378.4 g/mol, was obtained as 0.46 g, with 32% yield. Molecular structure was analyzed by means of IR and ^{1}H NMR: IR ((ν cm^{-1}) in KBr): $\nu_{C=O}$= 1700, ν_{imide}= 1650 , ν_{C-O}= 1080; ^{1}H NMR spectra (δ (ppm) in $CDCl_3 \backslash CF_3COOD$): δ_{Naph}= 8.76 (4H, s), δ_{N-CH2} = 4.20 (4H, t), δ_{C-CH2}= 1.74 (4H, m); 1.46 (4H, m); δ_{C-CH3}= 0.99 (6H, t)

Spectroscopic measurements

The electronic absorption spectra were measured using a Jasco V-530 UV/VIS spectrophotometer. Fluorescence emission spectra were recorded on a PTI-QM1 fluorescence spectrophotometer. Fluorescence quantum yields were calculated with respect to the absorption and fluorescence spectra of quinine sulfate in 0.5 M H_2SO_4 solution (φ_f = 0.546)[12,13]. The calculated relative fluorescence quantum yield was the value corrected for the refraction index differences between the sample and standard solutions[14]. The equation, used in calculations of fluorescence quantum yield was the following:

$$\varphi_{fu} = \varphi_{fr} \frac{S_U}{S_R} \frac{\left(1 - 10^{-D_R}\right)}{\left(1 - 10^{-D_U}\right)} \frac{n_U^2}{n_R^2} \tag{1}$$

where φ_f is the quantum yield, D is the absorbance at the excitation wavelength, S is the integrated emission band area, n is the solvent refractive index and U and R refer to the sample and reference (standard), respectively. All fluorescence measurements were conducted for dilute solutions in the absorbance range 0.1-0.15 at the excitation wavelength (concentrations 10^{-5}-10^{-6}mol·L^{-1}).

The fluorescence quenching measurements were monitored using a PTI-QM1 spectrometer.

Fluorescence quenching measurements

The quenching constants K_{SV} and k_q were calculated by means of Stern-Volmer relations[15].

$$I/I_0 = 1 + K_{SV} \cdot [Q] \tag{2}$$

$$K_{SV} = k_q \cdot \tau_f \tag{3}$$

where I_0 and I represent the fluorescence intensity of the fluorophore in the absence and presence of quencher molecules of concentration $[Q]$, τ_f is the radiative lifetime in the absence of the quencher.

Quenching of naphthalene, phenanthrene, pyrene and perylene fluorescence emission in acetonitrile solutions was studied with respect to increasing concentration of BBND. Concentration range of N,N′-bis-n-butyl derivative of naphthalene diimide was 0-1x10^{-2} M.

Cyclic voltammetry measurements

Cyclic voltammetry in chloroform was performed using a 3-electrode cell with a polished 2 mm glassy carbon as the working electrode, Pt wire as the counter electrode and Ag/AgCl as the reference electrode; the solution was 10^{-3} M in electroactive material and 0.1 M supporting electrolyte, tetrabutylammonium hexafluorophosphate ($TBAPF_6$). Voltammetry measurements were carried out using a Metrohm 694 VA stand and 693 VA processor. Cyclic voltammetry was performed using ferrocene as the internal reference electrode in the electrolyte solution of $TBAPF_6$ (0.1 M) in chloroform under nitrogen gas protection at a scan rate of 100 mV/s at 25 °C. All solutions were purged with nitrogen for at least 10 min before starting the measurements.

Results and Discussion

Absorption and emission properties

The absorption and fluorescence emission spectra of BBND in acetonitrile are shown in Figure 1. The remarkable mirror image relationship observed between the spectra should be noted, with a small Stokes shift (12 nm in acetonitrile). The absorption spectrum has an intense, short-wavelength band (A_{max} = 235 nm, not shown) and a structured band at lower energies with 3 maxima at 377, 357 and 340 nm, with absorption coefficients (ε) of 35200, 29250 and 17300 M^{-1} cm^{-1}, respectively. The emission spectrum shows 3 maxima, at 429, 407 and 390 nm, with an intensity ratio of 0.37:0.98:1. The excitation spectrum of the studied naphthalene diimide is identical to the absorption spectrum.

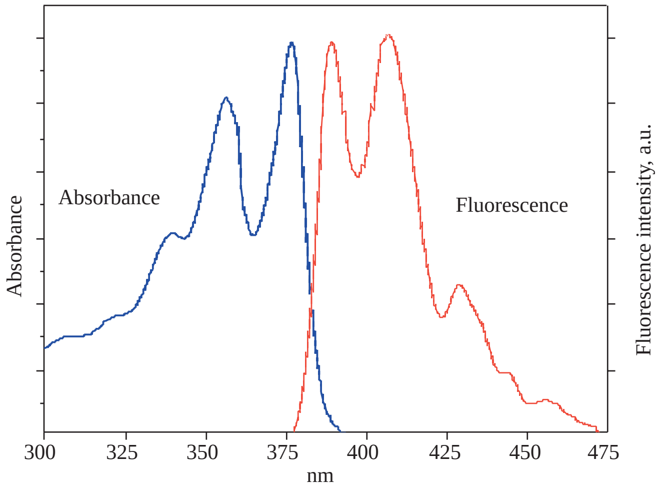


Figure 1. Absorption and emission (λ_{EXC}= 360 nm) spectra of N,N′-bis-n-butyl derivative, BBND, of naphthalene diimide in acetonitrile.

The value of the absorption coefficient of the studied compound, 3×10^4 M^{-1} cm^{-1} (see Table 1), allows the assignment of a π,π* character to the $S_0 \rightarrow S_1$ transition. The transition energy obtained from the intersection of the absorption and emission spectra is 312.6 kJ mol $^{-1}$. This value is lower than that for the analogous compound N-butyl-l,8-naphthalimide[17], where the transition energy for the first transition (π,π*) is 338.9 kJ mol $^{-1}$. The decrease in transition energy shows that the addition of a second imide group helps to stabilize the π,π* state, suggesting the presence of a strong π conjugation between the carbonyl groups and the naphthalene ring in BBND.

Table 1. UV-VIS spectroscopic data (A_{max} (nm) and ε (I·mol^{-1} cm^{-1})) of synthesized N,N′-bis-n-butyl derivative of naphthalene diimide, BBND, in acetonitrile.

λ_1^{abs}	ε_1	λ_2^{abs}	ε_2	λ_3^{abs}	ε_3
377	35200	357	29250	340	17300

λ_{1-3}^{abs} - are the positions of the maxima in the absorption spectrum (nm), ε - molecular extinction coefficient (L·mol^{-1} cm^{-1}).

The radiative lifetimes, τ_o, were calculated by the formula[18]τ_o= 3.5x10^8/ (υ_{max}^2x ε_{max}x $\Delta\upsilon_{1/2}$), where ν_{max} is the wavenumber in cm^{-1}, ε_{max} is the molar extinction coefficient at the selected absorption wavelength, and $\Delta\upsilon_{1/2}$ denotes the half-width of the selected absorption in cm^{-1}. Fluorescence lifetimes are estimated as τ_f= τ_o.Q_f and the rates of fluorescence as k_f = 1 / τ_o (Table 2).

Table 2. Spectral-luminescent data and photophysical parameters of synthesized N,N′-bis-n-butyl derivative of naphthalene diimide, BBND, in acetonitrile.

λ_1^{fl}	λ_2^{fl}	λ_3^{fl}	Q_f	τ_0	τ_f	k_f	E_s
390	407	429	0.002	8.2	16.4	1.22	312.6

*Here λ_{1-3}^{fl} - are the positions of the maxima in the fluorescence spectrum (nm), Q_f - fluorescence quantum yield, τ_0–radiative lifetime (ns), τ_f- fluorescence lifetime (ps), k_f - fluorescence rate constants, (10^8 s^{-1}), E_s– singlet energies (kJ·mol^{-1}).

Calculated fluorescence lifetimes are found to be 16 ps (Table 2). This value is in good correlation with the literature data: Green and Fox[19] report fluorescence lifetimes for naphthalene diimides at values lower than 20 ps.

The fluorescence of the studied BBND is rather weak, as shown by the low quantum yields (Table 2); fluorescence quantum yields are observed to be in the range 0.002-0.006 in acetonitrile.

Wintgens et al.[20] suggested the existence of an upper triplet n,π* state (T_2) close ($\Delta E = 8{,}368$ kJ mol $^{-1}$) to the singlet π,π* state of 1,8-naphthalimides. Green and Fox[19], on the other hand, could not detect any triplet level close to the singlet state for the dodecyl diimide (they found an S_1-T_1 gap of 113 kJ mol $^{-1}$), and proposed that the fast intersystem crossing in diimides could be promoted by vibrational coupling, or by means of an efficient spin-orbit coupling mechanism involving 4 carbonyl groups. However, the conclusions of Green and Fox[19] were based on triplet-triplet absorption experiments, and it cannot be ruled out that a $T_1 \rightarrow T_n$ absorption (with $n > 2$) was observed, with the T_2 level remaining undetected. In conclusion, the results described in a previous publication[11] and the results of this work support the idea that a rapid intersystem crossing process occurs for the deactivation of the singlet excited state of 1,4,5,8-naphthalenediimides. However, the mechanism of this process is not clear at present.

Electrochemistry of the BBND

Cyclic voltammometry (CV) is a valuable tool to study reversible redox behavior, electrochemical stability and to get information about LUMO energy values. The cyclic voltammogram of the compound is shown in Figure 2. Measured potentials and the calculated LUMO values are summarized in Table 3. The LUMO energy values were calculated based on the value of 4.8 eV for ferrocene (Fc) with respect to zero vacuum level[21,22]. BBND displays 2 cathodic peaks at -0.32 V and -0.81 V and two anodic peaks at -1.49 V and -1.06 V and 2 reversible two steps of reductions at -0.69 and -1.15 V (vs. Ag/AgCl) in $CHCl_3$ were calculated corresponding to the first and second electron processes from the imide groups (Figure1). The reduction potential with respect to ferrocene is –1.29 V and –1.75 V and LUMO is 3.51 eV. These results are comparable with those obtained by Ozser et al.[23] for 1,4,5,8-naphthalenediimide derivatives in chloroform solution, -1.21 V and -1.14 V, and –1.55 V and -1.71 V, and LUMO 3.59 eV and 3.66 eV, respectively.

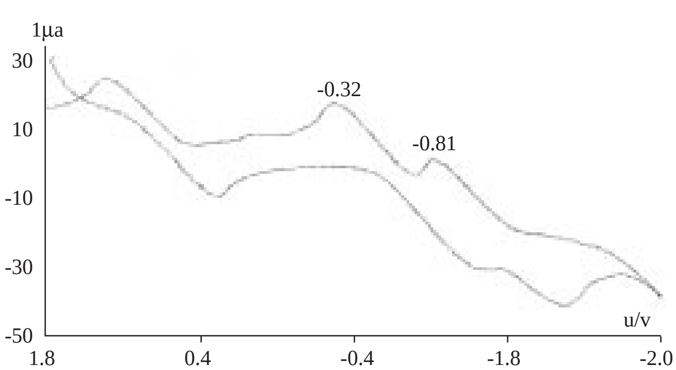


Figure 2. Cyclic voltammogram of N,N′-bis-n-butyl derivative of naphthalene diimide, BBND (in $CHCl_3$, supporting electrolyte 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$), scan rate 100 mVs^{-1}) at 25 °C.

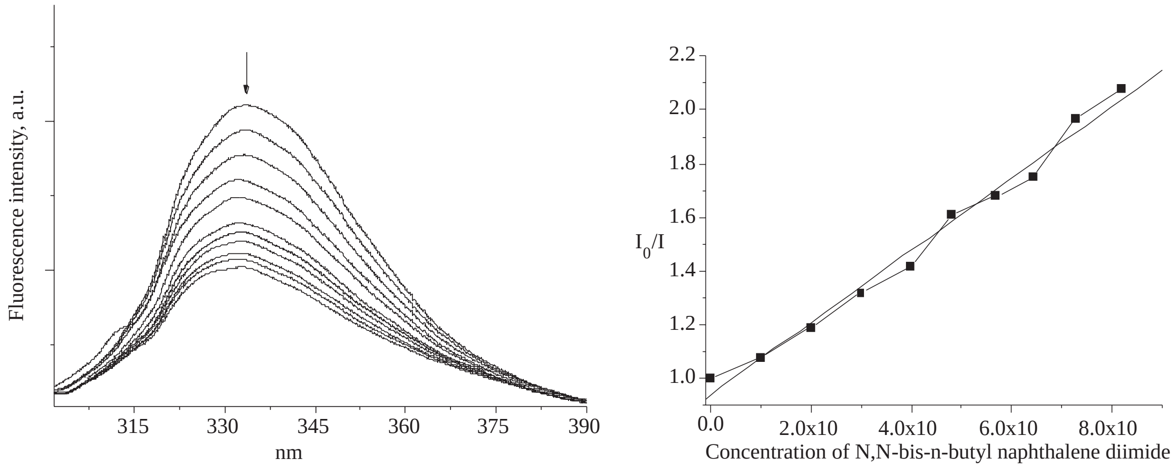
Table 3. Cyclic voltammetry ^{a} data and LUMO energy value of N,N′-bis-n-butyl derivative of naphthalene diimide, BBND, in chloroform. $E_{1/2}$/V vs. $F_c$is the reduction potential of BBND versus ferrocene electrode ($E_{1/2}$/V vs. F_c = ($E_{1/2}$/V vs. Ag/AgCl) – (E_{Fc}/V vs. Ag/AgCl)).

Electrode	E_{pc}/V	E_{pa}/V	$E_{1/2}$/V vs. Ag/AgCl	E_{Fc}/V vs. Ag/AgCl	$E_{1/2}$/V vs. F_c	LUMO/eV
Glassy Carbon	-0.32	-1.06	-0.69	0.60	-1.29	3.51
	-0.81	-1.49	-1.15	0.60	-1.75	

^{a}Supporting electrolyte, 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$). ^{b}Scan rate of 100 mVs^{-1}. [BBND]= 1 x 10^{-3} M.

Fluorescence quenching studies

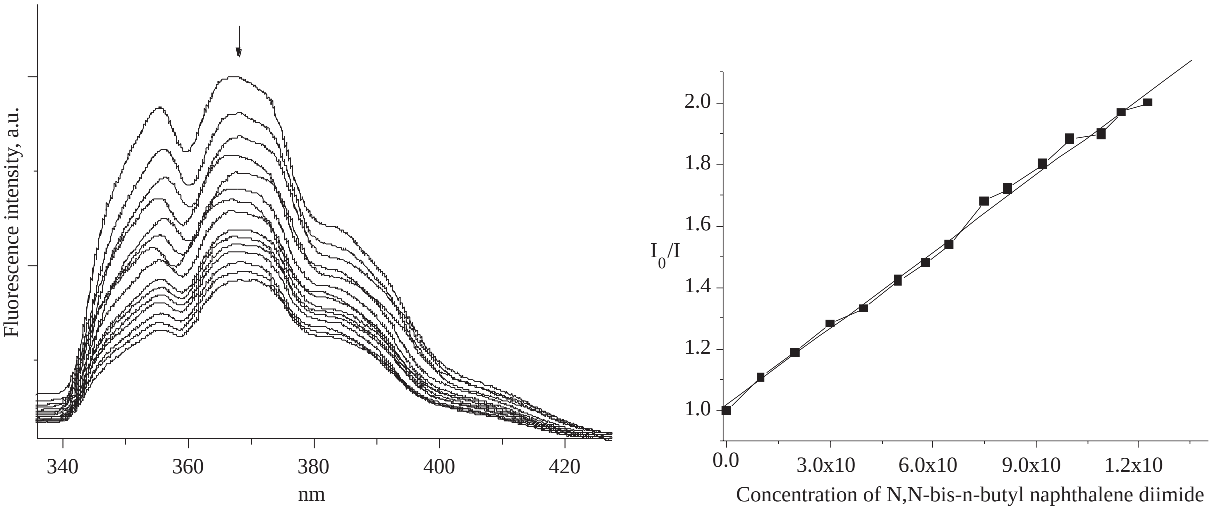
Naphthalene diimides are known to be electron acceptors[19]. BBND was found to quench the fluorescence emissions of aromatic donor molecules of naphthalene, phenanthrene, pyrene and perylene. Figures 3-6 show the quenching of fluorescence emissions of naphthalene, phenanthrene, pyrene and perylene by the addition of BBND and the corresponding Stern-Volmer plots. All spectra were corrected for inner and absorption effects before calculations. Calculated quenching rates are shown in Table 4. It is seen that the estimated quenching rates are in the diffusion limits for all the aromatic hydrocarbons.



(a) (b)

Figure 3. Fluorescence quenching of naphthalene by BBND in acetonitrile (A) and the corresponding Stern-Volmer plot (B).

It should be noted that the quenching rates of aromatic donor molecules in the presence of BBND are lower than the corresponding quenching rates in the case of quenching by perylene diimides. The rates of the quenching in the presence of perylene diimides were reported to have increased 5-fold from naphthalene to phenanthrene (from 4.4 x 10^{11} to 2.8 x $10^{12}M^{-1}$ s^{-1})[24] and 100-fold from phenanthrene to dihydrocarbazolo-carbazole (from 2.8 x 10^{12} to 2.5 x 10^{14} M^{-1} s^{-1})[25]. It was stated[25] that, probably, such high quenching rates observed for perylene diimide could be caused by static quenching in π-electron donor-acceptor complexes (perylene diimide-aromatic donor molecule) formed in the ground state.



(a) (b)

Figure 4. Fluorescence quenching of phenanthrene by BBND in acetonitrile (A) and the corresponding Stern-Volmer plot (B).

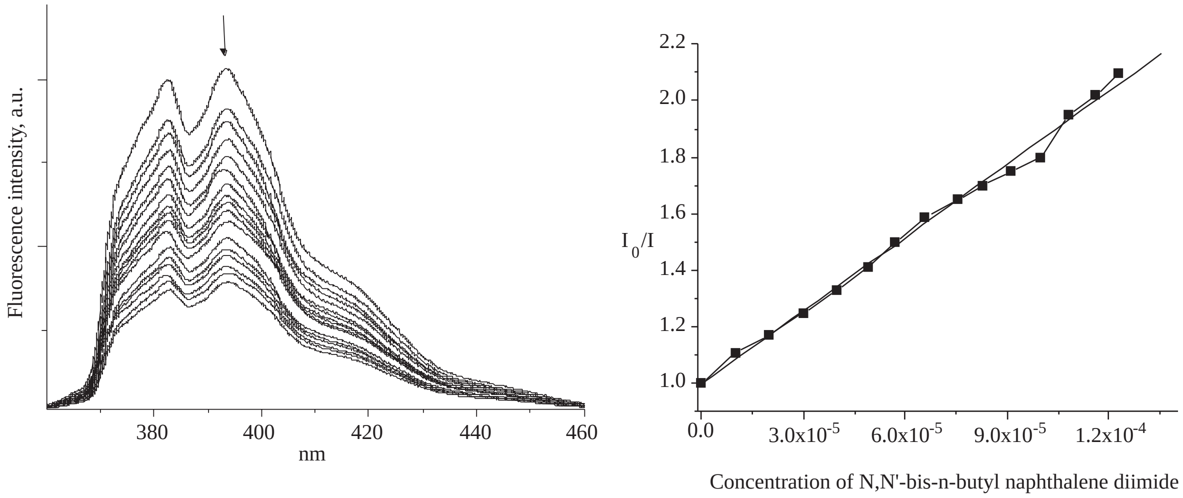


Figure 5. Fluorescence quenching of pyrene by BBND in acetonitrile (A) and the corresponding Stern-Volmer plot (B).

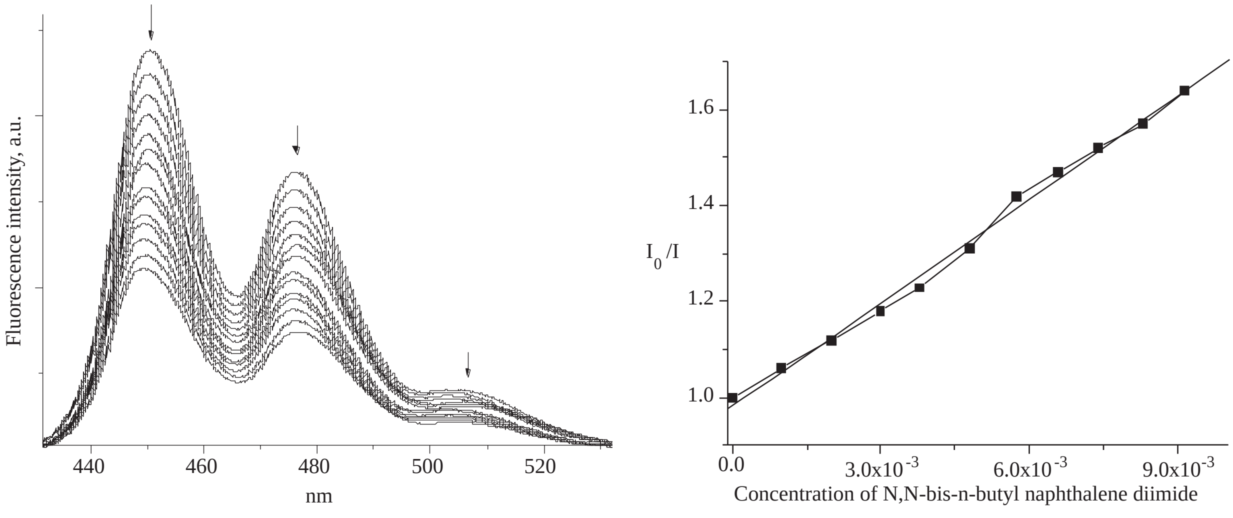
Thus, in contrast to perylene diimide derivatives, which form π-electron donor-acceptor complexes with aromatic donor molecules of naphthalene, phenanthrene, pyrene and perylene, BBND does not form π-electron-donor acceptor complexes with aromatic donor molecules.

Electron transfer and energy transfer processes could be responsible for the fluorescence quenching of the aromatic donor molecules in the presence of BBND.

The free-energy changes for oxidation of the donor and reduction of the acceptor via photoinduced electron transfer could be calculated from the Rehm-Weller equation[26]:

$$\Delta G_{EL-TR}(kJ/mol) = 96.486[E(D^{+}/D) - E(A/A^{-})] - E_{D}^{*} \qquad (4)$$

where the free-energy change for an encounter pair undergoing photoinduced electron transfer is computed by using redox potentials, and where $E^{*}_{D}$is the excitation energy of the excited state p articipating in quenching. Computed free energies of photoinduced electron transfer are presented in Table 4. In order for electron transfer to occur, ΔG_{EL-TR} has to be lower than –20.9 kJ/mol (the rule of thumb[27]).



(b)

Figure 6. Fluorescence quenching of perylene by BBND acetonitrile (A) and the corresponding Stern-Volmer plot (B).

Table 4. Fluorescence quenching rate constants of condensed aromatic electron donor molecules by the addition of N,N′-bis-n-butyl derivative of naphthalene diimide, BBND in acetonitrile.

Compound	K_{SV}	τ_0 (ns)	k_{qq} (M^{-1} s^{-1})	E_S^D (kJ·mol^{-1})	E (D^+/D) E (D^+/D) (volt)	ΔG_{EL-TR} (kJ·mol^{-1})	ΔG_{ENG-TR} (kJ·mol^{-1})
Naphthalene	1323.7	105*	1.3 10^{10}	384.9	2.60*	-42.0	-72.3
Phenanthrene	789.2	61*	1.3 10^{10}	345.4	2.44*	-17.9	-32.8
Pyrene	9356.9	475*	1.9 10^{10}	321.3	2.09*	-27.6	-8.7
Perylene	72.2	6*	1.2 10^{10}	274.9	1.67*	-21.7	37.7

Here *Ref. 18; K_{SV} – –Stern-Volmer constant of quenching; τ_0 – lifetime in the absence of quencher; k_{qq} – –diffusion constant of quenching; E_S^D- the excitation energy of the singlet excited state participating in quenching; E (D^+/D) – redox potential; ΔG_{EL-TR} and ΔG_{ENG-TR}– the free energy changes for electron and for energy transfer, correspondingly.

As seen in Table 4, ΔG_{EL-TR} values calculated for electron transfer from naphthalene, pyrene and perylene to the BBND are lower than the above calculated limit of –20.9 kJ/mol, which supports the ability of photo electron transfer. The ΔG_{EL-TR} value (-17.9 kJ/mol) calculated for the case of phenanthrene/BBND indicates that electron transfer between phenanthrene and the naphthalene diimide, BBND, is improbable; hence, in this case, fluorescence quenching probably occurs via energy transfer. This fact points to the possibility of electron transfer between the donor molecules and the studied naphthalene diimide, BBND.

The free-energy changes accompanying energy transfer either by electron exchange or by the dipole-dipole mechanism may be formulated as follows[28]:

$$\Delta G_{ENG-TR}(kJ/mol) = -E_{00}(D^{+}) + E_{00}(A^{+}) \qquad (5)$$

where the E_{00} – is the energy of 0–0 electronic transition.

Calculated free energies of energy transfer are presented in Table 4. The negative values of ΔG_{ENG-TR} for naphthalene, phenanthrene and pyrene point to the possibility of energy transfer between these aromatic molecules and BBND.

The positive value of ΔG_{ENG-TR} calculated for the case of perylene shows that the energy transfer process between perylene and the naphthalene diimide, BBND, is impossible; thus, the fluorescence quenching of perylene by BBND probably occurs solely via electron transfer.

Conclusions

The spectral-luminescent properties and electrochemistry of N,N′-bis-n-butyl derivative of naphthalene diimide, BBND, were studied. The main photophysical parameters (quantum yield, rate constants of fluorescence and of non-radiative deactivation and fluorescence lifetime) were determined.

The LUMO value (E_{LUMO}= –3.51 eV) was determined by cyclic voltammetry. BBND shows 2 reversible reduction steps at –1.29 V and –1.75 V vs. ferrocene, in chloroform.

Quenching of fluorescence emissions of aromatic donor molecules in acetonitrile has proven the electron acceptor ability of the BBND.

It has been found that, in contrast to perylene diimide derivatives, which form π-electron donor-acceptor complexes with aromatic donor molecules of naphthalene, phenanthrene, pyrene and perylene, BBND does not form π-electron-donor acceptor complexes with aromatic donor molecules.

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