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Effect of coaggregate formation on the fluorescence quenching of anthracene derivatives by *m-N,N*-diethylaminophenyl carboxylates with different chain lengths as quenchers

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ABSTRACT: In the dioxane–H_2O system, electron-transfer quenching processes have been observed between the excited 9-anthrylmethyl esters of butyric acid (**A-4**), caprylic acid (**A-8**), lauric acid (**A-12**) and palmitic acid (**A-16**) as fluorescence probes, and *m-N-N*-diethylaminophenyl esters of butyric acid (**P-4**), caprylic acid (**P-8**), lauric acid (**P-12**) and palmitic acid (**P-16**) as quenchers. The results indicate that the hydrophobic–lipophibic interaction (HLI)-driven coaggregation of an acceptor and a donor can very effectively facilitate the electron-transfer quenching process between the excited acceptor (or donor) and the ground-state donor (or acceptor) after they become preassociated inside the coaggregate species. Furthermore, the extent of HLI-driven coaggregation (preassociation) between the acceptor and the donor may be assessed from the slope B of the equation $I_0/I = A + B[\mathrm{Q}]$. The chain-length effect and the effect of solvent aggregating power were also observed. Copyright © 1999 John Wiley & Sons, Ltd.

KEYWORDS: coaggregate; anthracene derivatives; *m-N,N*-diethylaminophenyl carboxylates; fluorescence quenching

INTRODUCTION

Hydrophobic–lipophilic interactions (HLI) play an important role in chemical and biochemical processes such as conformational changes of biopolymers, the binding of a substrate to an enzyme, the formation of living cells from biological molecules, etc.[1–3] On the other hand, studies on photophysical and photochemical processes in organized assemblies have also attracted much attention.[4–6] Since aggregates of electrically neutral organic molecules are formed almost solely by HLI, some recent efforts have been dedicated to studies of hydrophobic effects on photochemical and photophysical processes.[7–9] It has been reported that aggregation, coaggregation and self-coiling of organic molecules affect the formation of excimers, the enhancement of energy transfer between excited donors and acceptors[9,10] and the hydrophobic acceleration of electron transfer processes.[11] In the present work, a novel finding is that at low concentrations ($\sim 10^{-6}$ M) ***A-*n*** can be used as an acceptor when **P-*n*** was used as a donor, but when **S-12** was used as an acceptor, ***A-12** becomes a donor in electron-transfer quenching process driven by HLI (see structures below). To our knowledge, these observations have not been reported previously. The fact that the critical coaggregate concentration (CoCAgC) value is so small may be considered a consequence of the high aggregating tendencies of the aggregators (Agrs).[1b] Clearly, HLI-driven processes such as self-aggregation and excimer formation on the one hand, and HLI-driven processes such as co-aggregation and electron transfer on the other, differ only in the fact that the former involves only one Agr, whereas the latter involves two different kinds of Agrs.

Electron transfer is a very active topic in chemical research,[12] such as the use of solar energy and photosynthetic process. It is also well known that medium effects, such as solvent polarity, microheterogeneous environment and the addition of salts, have an important influence on the behavior of photoinduced electron transfer between the donor and the acceptor in solution.[13] The present work broadens and extends studies on hydrophobic acceleration of electron-transfer processes facilitated by HLI-driven aggregation to the study of the electron-transfer process between excited 9-anthrylmethyl carboxylates (**A-*n*,*n*** = 4, 8, 12, 16) and *m-N,N*-diethylaminophenyl carboxylates (**P-*n*,*n*** = 4, 8, 12, 16) in order to demonstrate that aggregation can also exert a facilitating effect on the electron transfer process.

In the present work, excited 9-anthrylmethyl carboxy-

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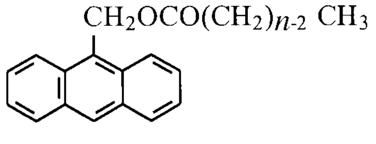
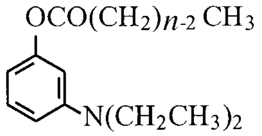
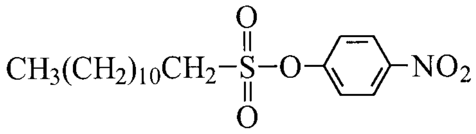


A-*n* (acceptor)
(*n* = 4, 8, 12, 16)



P-*n* (donor)
(*n* = 4, 8, 12, 16)

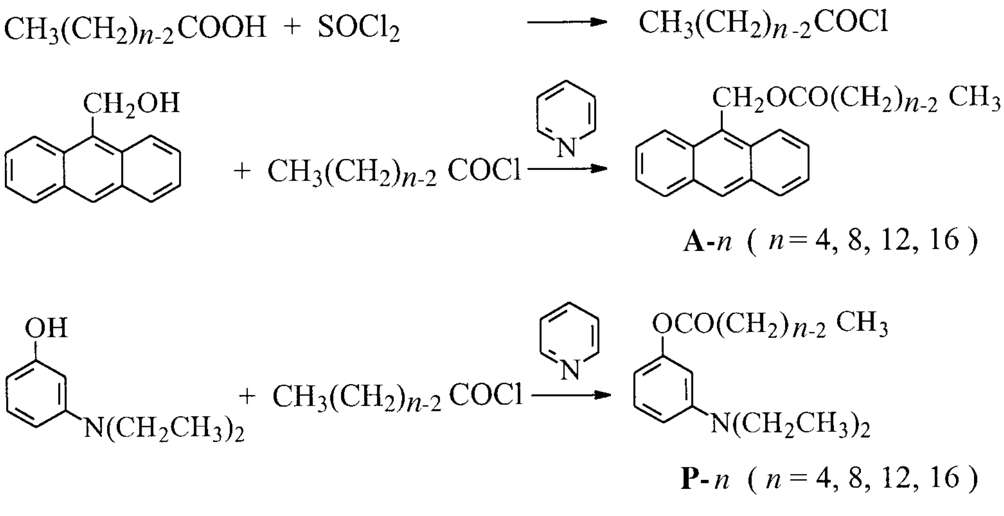


S-12

lates with different chain lengths (**A-*n*,*n*** = 4, 8, 12, 16) were used as acceptors and fluorescence probes, and *m*-*N*,*N*-diethylaminophenyl carboxylates (**P-*n*,*n*** = 4, 8, 12, 16) as donors and quenchers. HLI-facilitated electron-transfer processes between them were investigated by means of fluorescence spectroscopy in dioxane (DX)–H_2O systems with different Φ values, where Φ is the volume fraction of the organic component of an aqueous–organic mixture.

The occurrence of electron transfer between the excited acceptor and the ground-state donor results in fluorescence quenching of the excited acceptor **A-*n**** by **P-*n***. This is an expected result because it is well known that intermolecular electron transfer can occur between the lowest excited singlet state anthracene as the acceptor and *N*,*N*-diethylaniline as the donor.[12b] It is also well known that the electron-transfer process facilitated by HLI-driven aggregation is very similar to the static quenching process that occurs with fluorophores already associated with the quencher inside the coaggregate species.[14] We have now experimentally demonstrated for the first time that fluorescence quenching of **A-*n**** by **P-*n*** can be facilitated by HLI-driven coaggregation. Fluorescence can be quenched by electron transfer or energy transfer (or both electron and energy transfer). Quenching were carried out by exciting **A-*n*** at 360 nm with **P-*n*** (or **S-12**). The λ_{ex} value is larger than the **P-*n*** (or **S-12**) absorption wavelength. This indicated that the energy transfer from ***A-*n*** to **P-*n*** (or **S-12**) is endothermic, and the most probable quenching mechanism is electron transfer. In **A-*n*–P-*n*** systems, exciplex formation was not observed under our experimental conditions because of the polarity of the medium, as Weller[12b] and Ottolenghi[12c] have discussed previously.

Molecules may associate in solution for two reasons, by van der Waals force and by collision-induced weak charge-transfer (CT) interactions. However, both interactions are weak. On the other hand, previous studies have established that preassociation of chromophores driven by HLI can greatly facilitate excimer formation.[15] We speculate that enhancement of **A-*n**** fluorescence quenching might also be facilitated by HLI-driven coaggregation of **A-*n**** and **P-*n*** at very low concentrations (ca. 10^{-5} M). Furthermore, the efficiency of



Scheme 1

fluorescence quenching should depend on the chain length of the substitutent groups of the acceptor probes (**A-*n****) or the donor quenchers (**P-*n****) and on the solvent aggregating power (SAgP) of the reaction media. All these speculations have been found to be true.

EXPERIMENTAL

Apparatus. ^{1}H NMR spectra were obtained at 90 MHz on a Varian FX-90Q spectrometer with TMS as the internal standard. Chemical shifts are expressed in ppm (δ). UV–Vis spectra were recorded on a Perkin-Elmer 559 spectrometer.

Reagents and substrates. All target compounds were prepared in our laboratory and identified later by elemental analysis and ^{1}H NMR. All the esters were purified by flash column chromatography on silica gel with light petroleum–ethyl acetate as eluent.

Preparation of substrates. **A-*n*** and **P-*n*** were prepared by the reactions shown in Scheme 1.

A typical procedure for preparing the target compounds is as follows. A mixture of lauric acid and excess thionyl chloride in anhydrous benzene was stirred and refluxed for 3–4 h and excess thionyl chloride and benzene were removed under reduced pressure. A benzene solution of 9-anthracenemethanol (or 3-diethylaminophenol) and pyridine was added and stirred and refluxed for another 2–3 h. The solution was washed with saturated NaHCO$_3$ and saturated NaCl solution, dried with anhydrous Na$_2$SO$_4$ and the solvent was removed under reduced pressure. The product was purified by flash column chromatography on silica gel with light petroleum–ethyl acetate as eluent. The preparation of **S-12** has been reported elsewhere.[16]

*9-Anthrylmethyl ester of butyric acid (**A-4**).* Greenish liquid. ^{1}H NMR (CDCl$_3$), δ 1.1 (t, 3H), 1.8 (m, 2H), 2.5 (t, 2H), 6.2 (s, 2H), 7.4–8.6 (m, 9H); anal. calcd for C$_{19}$H$_{18}$O$_2$: C 81.99, H 6.52; found: C 81.57, H 6.52%; UV–Vis absorption, $\lambda_{\max}$ (ε) 347 nm (4500 l mol^{-1} cm^{-1}) 365 nm (6700 l mol^{-1} cm^{-1}) 385 nm (6200 l mol^{-1} cm^{-1}) (dioxane).

*9-Anthrylmethyl ester of caprylic acid (**A-8**).* Greenish liquid. ^{1}H NMR (CDCl$_3$), δ 0.8–1.7 (m, 13H), 2.3 (t, 2H), 6.1 (s, 2H), 7.4–8.3 (m, 9H), anal. calcd for C$_{23}$H$_{26}$O$_2$: C 82.60, H 7.84; found: C 82.74, H 7.82%; UV–Vis absorption: $\lambda_{\max}$ (ε) 347 nm (3200 l mol^{-1} cm^{-1}), 365 nm (4700 l mol^{-1} cm^{-1}), 385 nm (4300 l mol^{-1} cm^{-1}) (dioxane).

*9-Anthrylmethyl ester of lauric acid (**A-12**).* Greenish solid, m.p. 57–58 °C. ^{1}H NMR (CCl$_4$), δ 0.9–1.6 (m, 21H), 2.2 (t, 2H), 5.9 (s, 2H), 7.3–8.3 (m, 9H), anal. calcd for C$_{27}$H$_{34}$O$_2$: C 83.03, H 8.77; found: C 82.82, H 8.84%, UV–Vis absorption: $\lambda_{\max}$ (ε) 347 nm (4600 l mol^{-1} cm^{-1}), 365 nm (7000 l mol^{-1} cm^{-1}), 385 nm (6400 l mol^{-1} cm^{-1}) (dioxane).

*9-Anthrylmethyl ester of palmitic acid (**A-16**).* Greenish solid, m.p. 72–73 °C. ^{1}H NMR (CDCl$_3$): δ 0.9–1.7 (m, 29H), 2.3 (t, 2H), 6.1 (s, 2H), 7.4–8.4 (m, 9H), anal. calcd for C$_{31}$H$_{42}$O$_2$: C 83.36, H 9.48; found: C 83.38, H 9.64%; UV–Vis absorption: $\lambda_{\max}$ (ε) 347 nm (1200 l mol^{-1} cm^{-1}) 365 nm (1600 l mol^{-1} cm^{-1}) 385 nm (1400 l mol^{-1} cm^{-1}) (dioxane).

*m-N,N-Diethylaminophenyl ester of butyric acid (**P-4**).* Colorless liquid. ^{1}H NMR (CCl$_4$), δ 1.2 (m, 9H), 1.7 (m, 2H), 2.4 (t, 2H), 3.3 (q, 4H), 6.2–7.0 (m, 4H); anal. calcd for C$_{14}$H$_{21}$NO$_2$: C 71.46, H 8.99, N 5.95; found: C 71.47, H 9.17, N 6.06%; UV–Vis absorption, $\lambda_{\max}$ (ε) 260 nm (31 000 l mol^{-1} cm^{-1}), 304 nm (4600 l mol^{-1} cm^{-1}) (dioxane).

*m-N,N-Diethylaminophenyl ester of caprylic acid (**P-8**).* Greenish liquid. ^{1}H NMR (CCl$_4$), δ 1.0–1.8 (m, 19H), 2.4 (t, 2H), 3.3 (q, 4H), 6.2–6.9 (m, 4H); anal. calcd for C$_{18}$H$_{29}$NO$_2$: C 74.18, H 10.03, N 4.81; found: C 74.25, H 10.30, N 4.86%; UV–Vis absorption, $\lambda_{\max}$ (ε) 260 nm (25 000 l mol^{-1} cm^{-1}), 304 nm (3600 l mol^{-1} cm^{-1}) (dioxane).

*m-N,N-Diethylaminophenyl ester of lauric acid (**P-12**).* Greenish liquid. ^{1}H NMR (CCl$_4$), δ 0.9–1.8 (m, 27H), 2.4 (t, 2H), 3.3 (q, 4H), 6.3–6.9 (m, 4H); anal. calcd for C$_{22}$H$_{37}$NO$_2$: C 76.03, H 10.73, N 4.03; found: C 75.94, H 11.03, N 4.08%; UV–Vis absorption; $\lambda_{\max}$ (ε) 260 nm (41 000 l mol^{-1} cm^{-1}), 305 nm (7000 l mol^{-1} cm^{-1}) (dioxane).

*m-N,N-Diethylaminophenyl ester of palmitic acid (**P-16**).* Colorless solid, m.p. 34–35 °C. ^{1}H NMR (CCl$_4$), δ 0.9–1.8 (m, 35H), 2.4 (t, 2H), 3.4 (q, 4H), 6.3–7.0 (m, 4H); anal. calcd for C$_{26}$H$_{45}$NO$_2$: C 77.37, H 11.24, N 3.47; found: C 76.76, H 11.58, N 3.16%; UV–Vis absorption, $\lambda_{\max}$ (ε) 260 nm (50 000 l mol^{-1} cm^{-1}), 304 nm (7400 l mol^{-1} cm^{-1}) (dioxane).

RESULTS AND DISCUSSION

Previous work has shown that HLI can facilitate the electron-transfer process between excited 1-α-naphthyl-3-oxaalkanes and 2-alkyl-3,5,6-trichloro-1,4-benzoquinones in DX–H$_2$O systems with different Φ values.[14] In order to avoid the self-quenching of the fluorescence probe **A-*n*** and other competing photochemical processes, e.g. photoinduced dimerization of excited anthryl group, in our experiments on the hydrophobic acceleration of the electron-transfer process between **A-*n****** and **P-*n*,** the

Table 1. CAgC (10^{-6} M) of **A-*n*** (*n* = 4, 8, 12, 16) in DX–H_2O systems with different Φ values at 35 °C

	Φ					
	0.20	0.25	0.30	0.35	0.40	0.45
A-16					1.67	7.09
A-12			3.63	12.7		
A-8	4.89	15.0				
A-4	$\geq$8.64					

[a] Uncertainty is less than 10%.

concentration of **A-*n*** must be kept at less than its critical aggregate concentration (CAgC) values in the DX–H_2O solvent systems used, because it is generally accepted that below the CAgC, the **A-*n*** exists in its monomeric form in the DX–H_2O system. Therefore, the CAgCs of **A-*n*** were first measured by means of fluorescence spectroscopy in DX–H_2O systems with different Φ values,[1b,d] and the results are given in Table 1.

Examination of Table 1 reveals that, as expected, the CAgC values increase with increasing Φ values (or decreasing SAgP) for **A-*n*** in the DX–H_2O system, and the aggregating tendencies of **A- *n*** increase with increase in alkyl chain length.

From our experimental results in Figs 1 and 2 it is notable that different degrees of fluorescence quenching of all **A-*n***** fluorophores are observed when **P-*n*** or **S-12** is added.

It is very interesting that **A-12*** can be used as an acceptor when **P-*n*** is used as a donor, but when **S-12** is used as an acceptor, **A-12*** becomes a donor in the electron-transfer quenching process driven by HLI. This is consistent with the point of view that the terms electron donor and acceptor are only relative and a single species may behave both as a donor and as an acceptor in the study on organic electron-transfer complexes.[17]

In the fluorescence quenching experiments, the excitation wavelength used for **A-*n***** is 360 nm. It has been shown that in the presence of **P-*n*** in DX–H_2O solvent systems, **P-*n*** cannot be excited at 360 nm, and therefore it could not interfere with the fluorescence emission of **A-*n*****. Since the excitation wavelength (360 nm) is longer than the absorption wavelengths of **P-*n*** (260 and 304 nm) and **S-12** (270 nm), the energy transfer would be endothermic. This indicates that the possibility of energy transfer from ***A-*n*** to **P-*n*** (or **S-12**) could be excluded. Hence the most probable quenching mechanism is electron transfer, in harmony with previous reports that there is an electron-transfer process between excited anthracene and ground-state *N,N*-diethylaniline in non-polar solvents.[12b,d,e]

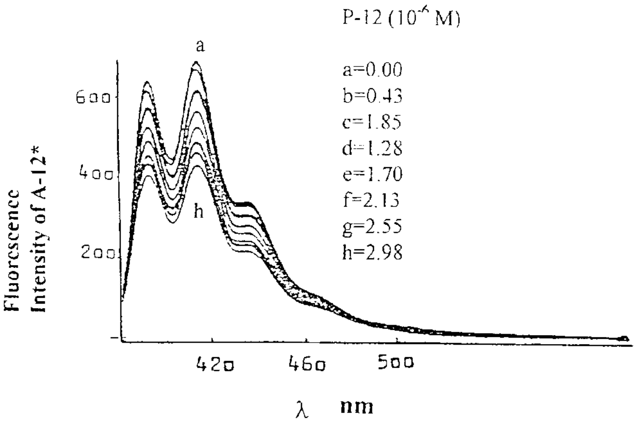


Figure 1. Effect of gradually increasing **P-12** concentration on the fluorescence intensity of **A-12** (1.0×10^{-6} M). $\phi = 0.30$; $\lambda_{ex} = 360$ nm; $t = 35$ °C

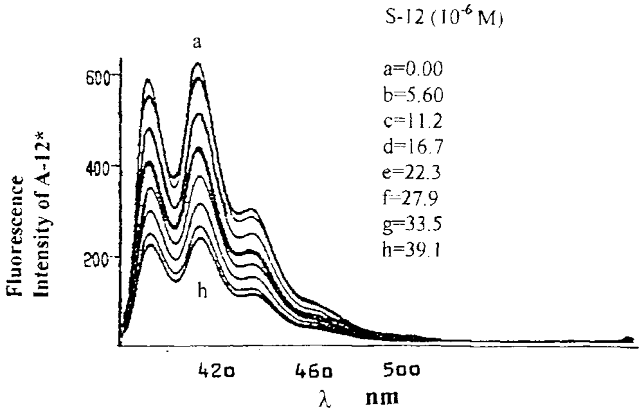


Figure 2. Effect of gradually increasing **S-12** concentration on the fluorescence intensity of **A-12** (1.0×10^{-6} M). $\phi = 0.30$; $\lambda_{ex} = 360$ nm; $t = 35$ °C

The fluorescence intensity of the excited fluorophore, **A-12***, decreases when the quencher **P-12** is added (see Fig. 1). In this case, **A-12*** was the electron acceptor and **P-12** was the electron donor, and HLI-driven preassociation precedes electron transfer between the excited acceptor and the ground donor inside the coaggregate species. In the case (Fig. 2) with **A-*n***** as the donor and **S-12** as the acceptor, the fluorescence intensity of **A-12*** also decreased quickly with increasing amount of **S-12** added to the DX–H_2O system, and hydrophobic acceleration of electron transfer is clearly demonstrated. However, in pure DX, there is no fluorescence quenching between **A-*n***** and **P-*n*** at low concentrations ($\sim 10^{-6}$ M), whereas in the DX–H_2O system there is fluorescence quenching between **A-*n***** and **P-*n*** at the same concentration, brought about by the proximity effect of the coaggregation under the influence of HLI. From the above experimental results, we may conclude that under different conditions, excited anthracene derivatives may serve either as electron acceptors or as electron donors. This observation has not been reported previously.

I_0/I vs [Q] plots show that there is a linear relationship [Eqn. (1)] between I_0/I and [Q] when [Q] is larger than or equal to CoCAgC, i.e. it is similar to the Stern–Volmer equation, i.e. $I_0/I = 1 + k_q \tau_0$ [Q]. In Eqn. (1), B is an

Table 2. *B* values (10^5 l mol^{-1}) of the quenching of **A-*n*** with **P-*n*** in DX–H_2O systems with different Φ values at 35 °C

	Φ						
	0.20	0.25	0.30	0.35	0.40	0.45	0.50
A-16 and **P-16**					3.51	1.04	0.470
A-12 and **P-12**			2.04	0.747	0.209	0.0718	0.0400
A-8 and **P-8**	0.213	0.0960	0.0357				
A-4 and **P-4**	/						

[a] Uncertainty is less than 10%.

Table 3. k_q (10^{12} l mol^{-1}s^{-1}) of the quenching of **A-*n*** with **P-*n*** in DX–H_2O systems with different Φ values at 35 °C

	Φ						
	0.20	0.25	0.30	0.35	0.40	0.45	0.50
A-16 and **P-16**					66	20	8.9
A-12 and **P-12**			38	14	3.9	1.4	0.75
A-8 and **P-8**	4.0	1.8	0.67				
A-4 and **P-4**	/						

[a] Uncertainty is less than 10% ($\tau_{An*} = 5.3$ ns).

empirical coefficient. Therefore, we used $B = k_q\,\tau_0$ for the evaluation of k_q, taking the τ_0 value as 5.3 ns for ***A-*n***.[18]

$$I_0/I = A + B[\mathrm{Q}] \qquad (1)$$

where I_0 is the fluorescence intensity of excited **A-*n*** in the absence of the quencher, *I* is the fluorescence intensity of excited **A- *n*** in the presence of the quencher, and [Q] is the concentration of the quencher.

Furthermore, in the **S-12–A-12** system, in Φ = 0.30 DX–H_2O at 35 °C, the *B* value is 5.15×10^4 and the corresponding k_q for **S-12** is 9.7×10^{12} l mol^{-1} s^{-1}. By comparing the aforesaid k_q or *B* value of **S-12** with the k_q value (3.8×10^{13} l mol^{-1} s^{-1}) or *B* value (2.04×10^5) of **P-12** in the ***A-12/P-12** system in Φ = 0.30 DX–H_2O (see Tables 2 and 3), we may conclude that **P-12** is better than **S-12** as a quencher in the ***A-12** fluorescence quenching process driven by HLI.

Certainly, a larger *B* value signifies more effective quenching of the excited acceptor (or the donor) by the donor (or the acceptor). The results indicate that the *B* values decrease with increasing Φ value (or decreasing SAgP) for the quenching of **A-12*** monomer by **P-12**, as expected. Different k_q values in solvents with different Φ value are summarized in Table 3.

Based on the assessed k_q values, which are much larger than 10^{10} l mol^{-1} s^{-1}, a diffusional control fluorescence quenching process appears to be very unlikely.[14] There-

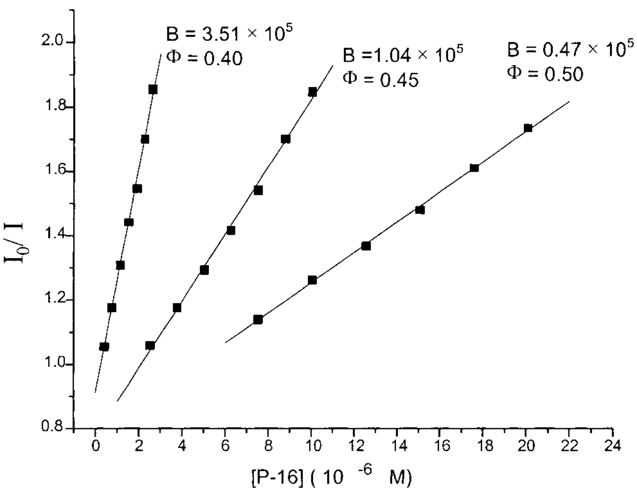


Figure 3. I_0/I of **A-16** (1.0×10^{-6} M) vs [**P-16**] in the DX–H_2O system with different Φ values at 35 °C

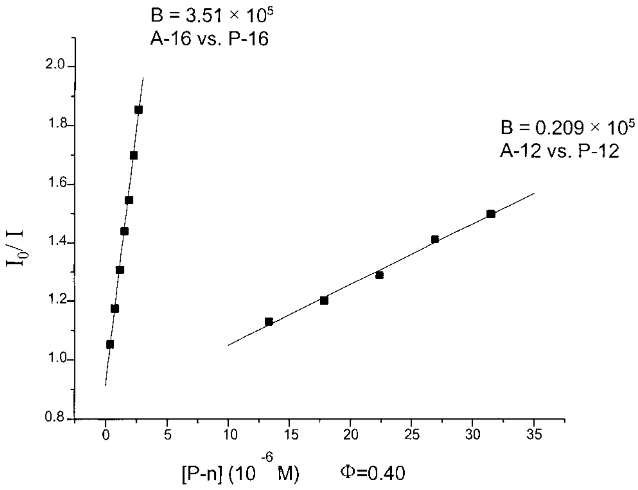


Figure 4. I_0/I of the probe (1.0×10^{-6} M) vs [**P-*n***] in the DX–H_2O system with Φ = 0.40 at 35 °C

fore, it might be concluded that the preassociation quenching process induced by HLI-driven aggregation is very similar to a static quenching process. The B values given in Figs 3 and 4 increase with decreasing Φ values, as expected from the fact that within a limited range of Φ values, SAgP and Φ are almost linearly related.[11] At larger Φ values, the tendency for aggregation and coaggregation of the organic aggregator will be smaller.

The I_0/I versus [Q] plots together with the corresponding B values are shown in Figs 3 and 4. They indicate that a larger B value signifies more effective fluorescence quenching by the quencher. Figure 4 shows that in the quenching process between **A-*n***** and **P-*n*,** there is a chain-length effect of the substitutent group, and Fig. 3 shows that there is an effect of SAgP on the efficiency of fluorescence quenching. On the other hand, on the basis of previous findings, the lifetime of the probe monomer is unchanged with increasing quencher concentration, i.e. it appears to be a constant within experimental uncertainty.[11,14] Therefore, the lifetimes of the probes were not measured. Finally, aggregation and coaggregation are consequences of stepwise processes.[19] For the measurement of CAgC or CoCAgC values based on property–concentration plots there is usually a transition region. Since measurements at these extremely low concentrations are not very precise, the intercept value at zero concentration may not be unity.

In conclusion, the above-mentioned results indicate that we have successfully integrated the study of photoinduced electron transfer processes with the study of aggregation of organic compounds. At low concentration ($\sim 10^{-6}$ M) in DX–H_2O systems the fluorescence quenching between **A-*n***** and **P-*n*** (or **S-12**) can be facilitated by the proximity effect of the coaggregation under the influence of HLI. Therefore, it is of importance to find an empirical parameter, herein designated B, for the fluorescence quenching process which may reflect the relative magnitudes of the tendencies of the fluorescence quenching in order to study structural effects on the donors and acceptors.

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