Articles

Electron-transfer fluorescence quenching processes in coaggregates between excited N-alkylcarbazoles as electron donors and 2, 4-dinitrophenyl carboxylates or pentafluorophenyl carboxylates as acceptors

SHI, Ji-Liang*(史济良)   YI, Hu-Nan(易湖南)   XU, Jia-Yi(许家移)   JIANG, Xi-Kui(蒋锡夔)

Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

Electron-transfer processes facilitated by hydrophobic-lipophilic interaction (HLI) between excited N-alkylcarbazoles (1-n, n = 4, 8, 12, 16) as electron donors and 2,4-dintrophenyl carboxylates (2-n, n = 4, 8, 12, 16) or pentafluorophenyl carboxylates (3-n, n = 4, 8, 12, 16) as electron acceptors have been investigated by means of fluorescence spectroscopy in aqueous or aquiorgano binary mixtures. The fluorescence quenching of -n* by 2-n or -n indicates that preassociation precedes the electron transfer. The extent of HLI-driven coaggregation of the acceptor and the donor may be assessed from the B value of the equation $I_0/I = A + B$ [Q]. The chain-length effect and possibly also a chain-foldability effect, as well as the solvent aggregating power (SAgP) effect have been observed. Comparison of the quenching constants (B) for 1-n*/2-n combinations and 1-n*/3-n combinations shows that the order of increasing B values for the quenching processes is 3-n < 2-n.

Keywords   Electron transfer process, HLI, fluorescence quenching

Hydrophobic-lipophilic interaction (HLI), together with other forces, can lead to the formation of aggregates (Ag's), micelles, and living cells from organic molecules in solvents with solvent aggregating power (SAgP).[1] Ag's of electrically neutral organic molecules are formed almost solely by HLI.[2-4] Molecular assemblies such as aggregates, vesicles, and micelles are being explored as artificial model systems for photophysical and photochemical studies.[5-7] It has been reported that aggregation, coaggregation, and self-coiling of organic molecules may facilitate excimer formation,[8,9] energy transfer,[10] or electron transfer.[11,12]

Photoinitiated electron transfer and related phenomena play an important role in life processes such as metabolism, photosynthesis, *etc.*, thus many researchers have looked for ways to study the basic chemistry of these processes in simplified model systems. It is well known that the effects of medium, polarity, surface charge of the micelle,[13] and nature of the head group of the surfactant,[13a,14] as well as chain-length of the alkyl group,[13a,15] all have important influences on the behavior of photoinduced electron transfer between the donor and the acceptor in solution. On the basis of our previous studies of hydrophobic acceleration of electron-transfer processes between carbon-containing compounds and p-nitrophenyl carboxylates,[11,12] we have now extended our research to those between nitrogen-containing heterocyclic donors and fluorine-containing acceptors.

In the present study, N-alkylcarbazoles (1-n) with different chain-lengths (n = 4, 8, 12, 16) were used as the electron donor and fluorescence probe, and 2,4-dinitrophenyl carboxylates (2-n) or pentafluorophenyl carboxylates (3-n) as the acceptor and quencher. We report some novel results of electron-transfer fluorescence quenching facilitated by HLI between excited 1-n and 2-n or 3-n in DX-H$_2$O system at low concentrations (~ 10^{-6} M).

There are three possible pathways leading to the fluorescence quenching of the excited-state fluorescence probe, namely, photoinitiated electron transfer, singlet-singlet energy transfer, and triplet-triplet energy trans-

fer. In the present work, the excitation wavelength of **1-**
n was 315 nm and there was no UV-Vis absorption for
2-n and **3-n** in the range of 250 ~ 300 nm, that is,
there was no possibility of energy transfer and the path-
way leading to the fluorescence quenching of **1-n** * is
photoinitiated electron transfer. The HLI driven electron
transfer between the donor and acceptor has been found
to be very similar to the static quenching process, which
is characterized by an invariant fluorescence life-
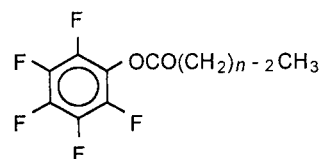time.[11,16,17] We have found that the extent of HLI-driven

coaggregation between the donor and the acceptor can be
assessed from the empirical slope value (B) of the e-
quation $I_0/I = A + B [Q]$, and the fluorescence
quenching depends on the chain length of the substitu-
tent groups of the quenchers or the probes and on the
SAgP of the reaction media.[12] B values of the **1-n/2-n**
pairs have been compared with those of the **1-n/3-n**
pairs in order to assess the relative effectiveness of the
quenchers **2-n** or **3-n** .
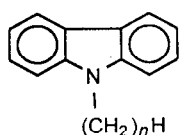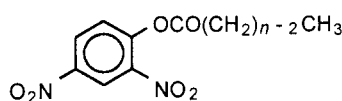
1 - n (donodrs)   2 - n (acceptors)   3 - n (acceptors)
n = 4, 8, 12, 16   n = 4, 8, 12, 16   n = 4, 8, 12, 16

Experimental

Apparatus   ¹H NMR spectra were obtained at
90 MHz on a Varian FX-90Q spectrometer with TMS as
the internal standard and ¹⁹F NMR on a Varian EM
360L spectrometer with CF₃COOH as the external stan-
dard. Chemical shifts are expressed in ppm (δ) . Flash

column chromatography was performed on silica gel with
petroleum ether-ethyl acetate as the eluent.

Reagents and the preparation of the target compounds

All target compounds were prepared by reactions
shown in Scheme 1 in this laboratory and identified later

Scheme 1
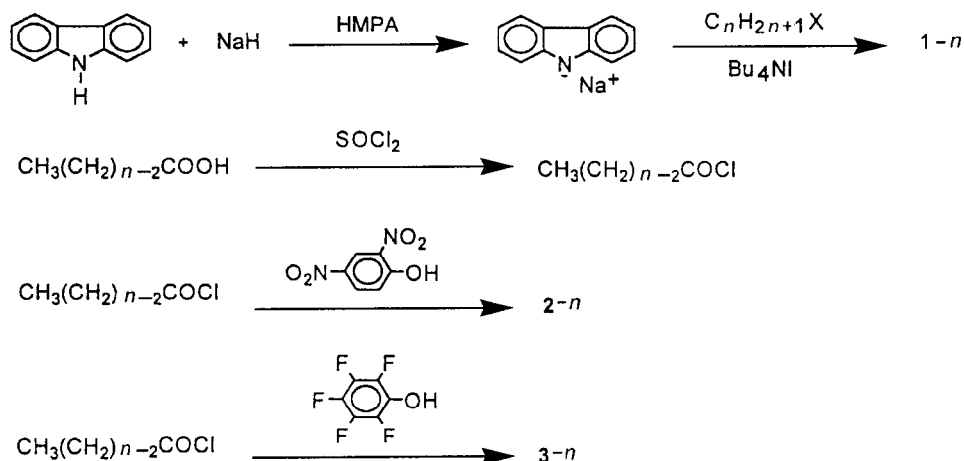
by elemental analysis, ¹H NMR, and ¹⁹F NMR spectra.
2-n and **3-n** are new compounds, **1-n** has been report-
ed.[18b] They were purified by flash column chromatogra-
phy on silica gel with petroleum ether-ethyl acetate as

the eluent. They possess UV-Vis, the strongest absorp-
tion λ_{max} at 233 ± 2 nm in cyclohexane for **2-n** and λ_{max}
at 198 ± 2 nm in cyclohexane for **3-n** respectively.

2,4-Dinitrophenyl carboxylates (**2-n**) and penta-fluorophenyl carboxylates (**3-n**) were prepared by the method of this laboratory.[18a]

2,4-*Dinitrophenyl butylate* (**2-4**)   Yellowish solid, mp: 40 ~ 41℃. λ(max)(cyclohexane): 233 nm. δ_H(CDCl$_3$): 1.07(t, $J = 7.5$ Hz, 3H), 1.47 ~ 2.00(m, 2H), 2.67(t, $J = 7.5$ Hz, 2H), 7.43(d, $J = 9.0$ Hz, 1H), 8.47(dd, $J = 9.0$, 3.0 Hz, 1H), 8.92(d, $J = 3.0$ Hz, 1H). Anal. C$_{10}$H$_{10}$N$_2$O$_6$. Cal cd.: C, 47.24; H, 3.93; N, 11.02. Found: C, 47.47; H, 3.84; N, 11.01.

2,4-*Dinitrophenyl octylate* (**2-8**)   Yellowish liquid. λ(max)(cyclohexane): 233 nm. δ_H(CDCl$_3$): 1.10 ~ 1.25(m, 3H), 1.35 ~ 1.75(m, 8H), 1.75 ~ 2.15(m, 2H), 2.78(t, $J = 7.5$ Hz, 2H), 7.52(d, $J = 9.0$ Hz, 1H), 8.60(dd, $J = 9.0$, 3.0 Hz, 1H), 9.02(d, $J = 3.0$ Hz, 1H). Anal. C$_{14}$H$_{18}$N$_2$O$_6$. Calcd.: C, 54.19; H, 5.81; N, 9.03. Found: C, 54.07; H, 5.82; N, 8.79.

2,4-*Dinitrophenyl dodecylate* (**2-12**)   Yellowish solid, mp 45 ~ 46℃. λ(max)(cyclohexane): 233 nm. δ_H(CDCl$_3$): 0.84 ~ 1.00(m, 3H), 1.04 ~ 1.54 (m, 16H), 1.54 ~ 1.93(m, 2H), 2.67 (t, $J = 7.5$ Hz, 2H), 7.43(d, $J = 9.0$ Hz, 1H), 8.45(dd, $J = 9.0$, 3.0 Hz, 1H), 8.92(d, $J = 3.0$ Hz, 1H). Anal. C$_{18}$H$_{26}$N$_2$O$_6$. Calcd.: C, 59.01; H, 7.10; N, 7.65. Found: C, 58.91; H, 7.28; N, 7.57.

2,4-*Dinitrophenyl hexadecylate* (**2-16**)   Yellowish solid, mp 59 ~ 61℃. λ(max)(cyclohexane): 233 nm. δ_H(CDCl$_3$): 0.85 ~ 1.15(m, 3H), 1.25 ~ 1.70(m, 24H), 1.75 ~ 2.05(m, 2H), 2.78(t, $J = 7.5$ Hz, 2H), 7.53 (d, $J = 9.0$ Hz, 1H), 8.62(dd, $J = 9.0$, 3.0 Hz, 1H), 9.03(d, $J = 3.0$ Hz, 1H). Anal. C$_{22}$H$_{34}$N$_2$O$_6$. Calcd.: C, 62.56; H, 8.06; N, 6.64. Found: C, 62.74; H, 8.25; N, 6.62.

Pentafluorophenyl butylate (**3-4**)   Colorless liq uid. λ(max)(cyclohexane): 198 nm. δ_H(CDCl$_3$): 1.07(t, $J = 7.5$Hz,3H), 1.26 ~ 2.06(m,2H), 2.68 (t, $J = 7.5$Hz,2H). δ_F(CDCl$_3$):76.23(d, $J = 20.7$ Hz, 2F), 81.80(t, $J = 20.7$ Hz, 1F), 85.80(t, $J = 20.7$ Hz, 2F). Anal. C$_{10}$H$_7$O$_2$F$_5$. Calcd.: C, 47.24; H, 2.75; F, 37.40. Found: C, 46.70; H, 2.50; F, 37.53.

Pentafluorophenyl octylate (**3-8**)   Colorless liq uid. λ(max)(cyclohexane): 198 nm. δ_H(CDCl$_3$): 0.75 ~ 1.08(m, 3H), 1.15 ~ 1.60(m, 8H), 1.60 ~ 2.00(m, 2H), 2.7(t, $J = 7.5$ Hz, 2H). δ_F(CDCl$_3$):

77.67(d, $J = 20.7$ Hz, 2F), 82.77(t, $J = 20.7$ Hz, 1 F),87.07(t, $J = 20.7$Hz,2F). Anal. C$_{14}$H$_{15}$O$_2$F$_5$. Calcd.: C, 54.19; H, 4.84; F, 30.65. Found: C, 53.55; H, 4.75; F, 30.83.

Pentafluorophenyl dodecylate (**3-12**)   White solid, mp 27.5 ~ 28 ℃. λ(max)(cyclohexane): 198 nm. δ_H(CDCl$_3$): 0.59 ~ 2.29 (m, 21H), 2.66(t, $J = 7.5$ Hz, 2H). δ_F(CDCl$_3$): 75.18(d, $J = 19.2$ Hz, 2F), 80.78(t, $J = 19.2$ Hz, 1F), 85.00(t, $J = 19.2$ Hz, 2F). Anal. C$_{18}$H$_{23}$O$_2$F$_5$. Calcd.: C, 59.01; H, 6.28; F, 25.96. Found: C, 59.03; H, 6.45; F, 25.57.

Pentafluorophenyl hexadecylate (**3-16**)   White solid, mp 50.5 ~ 51.8℃. λ(max)(cyclohexane): 198 nm. δ_H(CDCl$_3$): 0.72 ~ 1.07(m, 3H), 1.12 ~ 2.12 (m, 26H), 2.65(t, $J = 7.5$ Hz, 2H). δ_F(CDCl$_3$): 76.20(d, $J = 20.7$ Hz, 2F), 81.70(t, $J = 20.7$ Hz, 1F), 85.90(t, $J = 20.7$ Hz, 2F). Anal. C$_{22}$H$_{31}$O$_2$F$_5$. Calcd.: C, 62.56; H, 7.35; F, 22.51. Found: C, 62.60; H, 7.50; F, 22.46.

Electronic spectra   All aquiorgano solutions used for spectroscopic measurements were prepared from deionized water, and dioxane (DX) was purified by a standard procedure.[20] Fluorescence emission spectra of nondegassed aquiorgano solutions (DX-H$_2$O) of CBZ-n were recorded by a Perkin-Elmer LS-50 spectrometer at 35℃. In the fluorescence intensity measurements, an excitation wavelength of 315 nm was used for **1**-n, and the emission maximum is 353 nm for the excited monomer. Furthermore, the wavelengths of fluorescence peaks in the emission of excited **1**-n were red shifted as compared with the value in Ref. 21 because of the po-larity of DX-H$_2$O solution. The error in the fluorescence intensity measurements was less than ± 5%. UV-Vis spectra were recorded on a Perkin-Elmer 559 spectrome-ter.

Results and discussion

It has been reported that HLI-driven coaggregation facilitates electron-transfer processes between electron donors and acceptors in aquiograno binary mixtures.[12,18a] In order to avoid the self-quenching of the fluorescence probe **1**-n by forming excimers with itself, it must be kept at the monomeric state in the solvent systems used. It is generally accepted that below CAgC, the **1**-n exists in its monomeric form in the DX-H$_2$O system. There-

fore, the critical aggregate concentrations (CAgC) values of 1-n were first measured by previous reported method,[22] the CAgC values of 1-n in DX-H₂O system of

graded Φ values are listed in Table 1, where Φ represents the volume fraction of the organic component of the aquiorgano mixtures. Furthermore, it has been experi-

Table 1 CAgC values (10^{-7} M) of **1** in DX-H₂O system of different Φ values at 35 ℃ [18b]

Agr	Φ (DX-H₂O)
	0	0.20	0.25	0.30	0.35	0.40	0.45
1-4	22.6 ± 1.74						
1-8		8.00 ± 0.78	26.0 ± 0.89				
1-12				3.17 ± 0.31	9.06 ± 0.33		
1-16						7.17 ± 0.76	16.8 ± 0.05

mentally demonstrated that in the absence of 1-n , there is no fluorescence from 2-n or 3-n when the system is excited by an excitation wavelength of 315 nm, in other words, in the presence of 1-n, the fluorescence actually originate from excited 1-n*, and there is no interference of this fluorescence by the presence of 2-n or 3-n. To illustrate, Fig. 1 shows that the fluorescence intensity of the excited fluorescence probe 1-12* decreases with the increasing concentration of the quencher 2-12 in the $\Phi = 0.30$ DX-H₂O system. When 3-12 was used as the quencher, the same phenomenon as that of Fig. 1 was also observed. These results clearly demonstrate that quenching of the fluorescence of excited 1-n* by 2-n or 3-n has been observed. We have found that an empirical B value can be evaluated by plotting I_0/I against the concentrations of the quencher, where I_0 and I represent the fluorescence intensity of the excited donor in
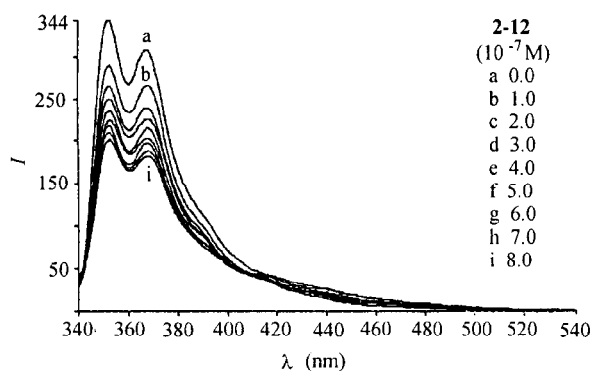
[Q] is the concentration of the quencher. Plots of I_0/I vs. [Q] show that there is a linear relation with slope B (eq. 1) between I_0/I and [Q] when [Q] is larger than or equal to its CoCAgC.

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

Results of these B value measurements at graded Φ values of DX-H₂O systems are summarized in Table 2 and Table 3.

Table 2 B Values (10^5 L/mol) for the fluorescence quenching of 1-n* of different chain-lengths by 2-n in aqueous or DX-H₂O of different Φ values at 35 ℃ [a,b]

	\multicolumn{2}{c}{1-4}	\multicolumn{2}{c}{1-8}	\multicolumn{2}{c}{1-12}	\multicolumn{2}{c}{1-16}
Φ	0	0.20	0.25	0.30	0.35	0.40	0.45
2-4	0.60						
2-8	3.26	4.23*	2.06*	5.89*	1.10*	5.94*	1.10*
2-12	8.00	7.57	1.86	7.22	2.37	5.40	1.70
2-16	9.64	12.9	4.63	18.9	4.50	6.35*	3.00

a. The uncertainty of B values is less than ± 10%.

b. Normally the concentration is 2.0×10^{-7} M for 1-n. Asterisked B values were measured at [1-n] of 1.0×10^{-7} M.

Fig. 1 Concentration effect of 2-12 on the fluorescence intensity of 1-12* (2×10^{-7} M) in the $\Phi = 0.30$ DX-H₂O system at 35 ℃.

	2-12
	(10^{-7} M)
a	0.0
b	1.0
c	2.0
d	3.0
e	4.0
f	5.0
g	6.0
h	7.0
i	8.0

Fig. 2 illustrates the fluorescence quenching of 1-8* by 2-16 and 3-16 in DX-H₂O system of $\Phi = 0.20$, the B values can be evaluated from the linear plotsof I_0/I vs. the concentration of the quencher. Fig. 2 shows the I_0/I against concentration of 2-16 and 3-16 plots at a fixed concentration of 1-8 [2×10^{-7} M] in $\Phi = 0.20$ DX-H₂O system. The B values derived from the slopes of these plots are $12.9 \times 10^5 \, M^{-1}$ for the 1-8/2-16 combination and $3.25 \times 10^5 \, M^{-1}$ for the 1-8/3-16 (see Table 2, 3). A larger B value signifies a more effective fluorescence quenching of the excited donor by the acceptor.

the absence and presence of quencher respectively, and

The results indicate that the order of decreasing B values for the quenching of 1-8* monomer by 2-16 and 3-16 is 2-16 > 3-16. At this juncture, we can make a comparison of Table 2 and Table 3, and find out that the fluorescence quenching rate of 1-n* in the presence of 2-n and 3-n with the same number of carbon atoms to be 2-n > 3-n. But, in the comparison B values of the combination 1-12/2-8 with 1-12/3-8 in the $\Phi = 0.35$ DX-H_2O system, and 1-16/2-8 with 1-16/3-8 in the $\Phi = 0.45$DX-H_2O system, their B values are rather close to each other within experimental uncertainty because there might be the effects of self-coiling for 1-12 or 1-16, and at larger Φ values, the difference of the coaggregation tendencies of 2-8 and 3-8 was also small. It is interesting to note that the difference of B values between 2-n and 3-n is dependent on the Φ value.

Table 3 B Values (10^5 L/mol) for the fluorescence quenching of 1-n * of different chain-lengths by 3-n in aqueous or DX-H_2O systems of different Φ values at $35 \,^{\circ}C$ [a,b]

	1-4	1-8		1-12		1-16	
Φ	0	0.20	0.25	0.30	0.35	0.40	0.45
3-4	0.48						
3-8	2.64	1.90	0.60	4.68*	1.30*	3.89*	1.33*
3-12	3.55	4.26*	1.26*	4.50*	2.14*	4.04*	0.72*
3-16	3.76	3.25	1.14	5.80*	3.67	4.30	0.96

a. The uncertainty of B values is less than ± 10%.

b. Normally the concentration is 2.0×10^{-7} M for 1-n. Asterisked B values were measured at [1-n] of 1.0×10^7 M.

Fig. 3 shows that there is a chain-length effect of the quencher 2-n on the B values of the quenching processes in coaggregates. The B value increases with the increase of the atom number in the chain from the 2-4 to 2-16. The increasing order for B values of 1-4* quenched by 2-n is 2-4 < 2-8 < 2-12 < 2-16. Similarly, as shown in Table 3, the comparison of B values for 1-4/3-n combinations in the $\Phi = 0$ DX-H_2O system shows the same chain-length effect. It shows that molecules with longer hydrocarbon chains have a larger coaggregating tendency. Furthermore, a chain-length effect of fluorescence probe on the B values can also be observed, as shown by Fig. 4, *i.e*., the B value increases with the chain-length of fluorescence probe 1-n. In other words, a fluorescence probe of longer chain-length has a greater B value in its quenching by the same acceptor 2-12.
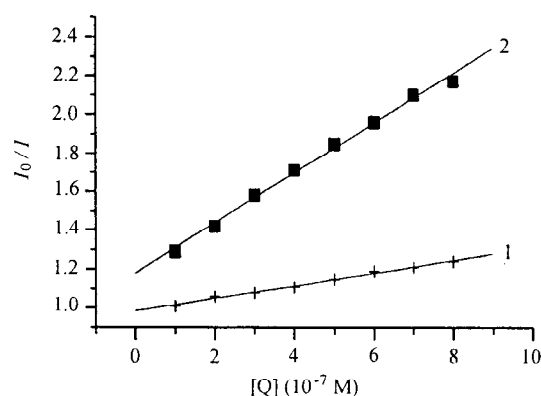

Fig. 2 I_0/I plots against the concentration of the quenchers for the quenching of 1-8* by 2-16 (curve 2), and 3-16 (curve 1) in the $\Phi = 0.20$ DX-H_2O system at $35 \,^{\circ}C$. The concentration of 1-n is 2×10^7 M.

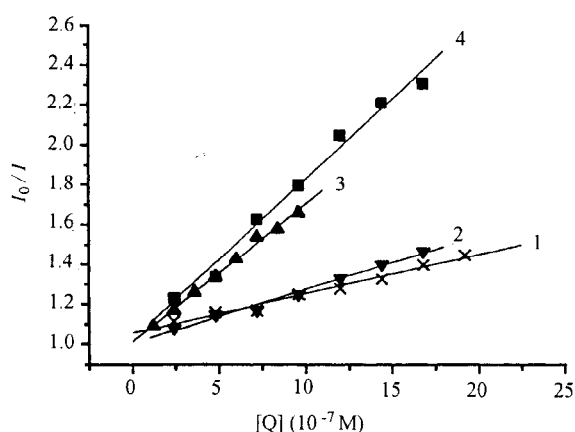**Fig. 3** I_0/I plots against the concentration of the quenchers for the quenching of 1-4* by 2-4 (curve 1), 2-8 (curve 2), 2-12 (curve 3), and 2-16 (curve 4) in H_2O at $35 \,^{\circ}C$. The concentration of 1-4 is 5×10^7 M.

The order of B values is 1-8 < 1-12 < 1-16 in the $\Phi = 0.40$ system at $35 \,^{\circ}C$. More interestingly, examination of Table 3 also reveals that there may be a chain-foldability effect[3, 23, 24] on B values, *i.e*., the B values for the 1-8/3-16 combination is lower than those for the 1-8/3-12 combination in the $\Phi = 0.20$ and also in the $\Phi = 0.25$ system, that is, flexible hydrocarbon chains with more than about 12 CH_2s have a greater tendency to fold on itself than those with 12 CH_2s, and consequently, the co-

aggregating tendency of a 16-carbon chain becomes smaller than that of the 12-carbon chain.

In a given aquiorgano solvent system, the SAgP, an inherent solvent property, can be raised by decreasing the Φ values. It is expected that the lower the Φ value, the greater should be the B value of the fluorescence quenching of the excited donor. Comparison of the results from Table 2 and Table 3 shows that the expected results have been found. In Table 2, for example, the B value (7.57×10^5 L/mol) for the fluorescence quenching of 1-8* by 2-12 in the $\Phi = 0.20$ DX-H_2O solvent system is greater than the value (1.86×10^5 L/mol) in the $\Phi = 0.25$ system. In Table 3, the B value (4.26×10^5 L/mol) for the fluorescence quenching of 1-8* by 3-12 in the $\Phi = 0.20$ DX-H_2O solvent system is greater than the value (1.26×10^5 L/mol) in the $\Phi = 0.25$ system. These observations are good examples of the SAgP effect on the aggregation and coaggregation behavior between organic molecules in a given solvent system. In an aquiorgano binary solvent system of lower Φ value, the fluorescence probe and the quencher have a greater coaggregation tendency, and thus have a larger B value for the electron-transfer quenching process.
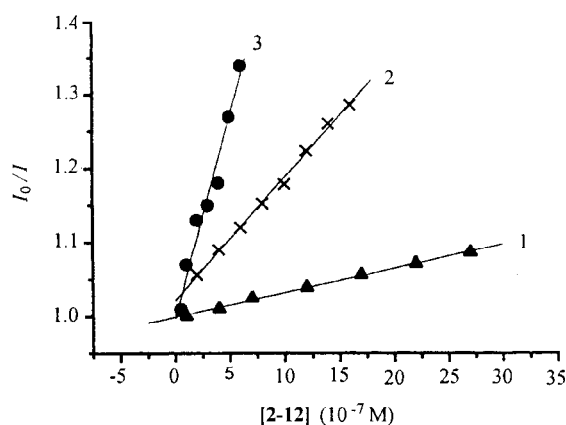

Fig. 4 I_0/I plots against the concentration of 2-12 for the quenching of 1-8* (curve 1, $B = 0.33 \times 10^5$ L/mol), 1-12* (curve 2, $B = 1.69 \times 10^5$ L/mol), and 1-16* (curve 3, $B = 5.40 \times 10^5$ L/mol) by 2-12 in the $\Phi = 0.40$ DX-H_2O system at 35°C. The concentration of 1-n is 2×10^7 M.

It has been reported[12] that the quenching rate of fluorescence in electron-transfer fluorescence quenching process in coaggregate is much larger than the diffusion-limited quenching rate (10^{10} M^{-1} S^{-1}).

We conclude that the B values for the electron-transfer quenching process in coaggregates depend on the chain-length of the fluorescence probe and the quencher, and on the SAgP of the media, and, there may also be a chain-foldability effect on it. In other words, B values increase with increasing HLI. Therefore, coaggregation brought about by HLI between organic molecules can very effectively accelerate the electron-transfer process between the excited donor and acceptor preassociated in coaggregates.

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(JIANG, X.H.; DONG, L.J.)