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# Spectrophysics of Coumarin-Based Chromophore

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**Abstract :** In this work, we comprehensively explore the spectral and photophysical properties of a coumarin-based dye (**1**) in neat solvents. The modulation of Stokes shifts, emission quantum yields ( $\Phi_F$ ) and excited-state lifetimes of **1** by local environment (polarity, polarizability, viscosity and hydrogen bonding) signifies the formation of intramolecular charge state (ICT) from the amino group to the coumarin moiety. Collectively, in the more viscous polar solvents the rotation of the amino group is restricted, exponentially decreasing the non-radiative rate constants ( $k_{nr}$ ).

**Keywords :** SPECTROPHYSICS, INTERMOLECULAR CHARGE TRANSFER, NON-RADIATIVE RATE CONSTANT, EXCITED-STATE LIFETIME, LOCAL ENVIRONMENT.

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## Introduction

Organic molecules' fluorescence is heavily reliant on their surroundings.[1] The bulk properties of a fluorophore or a chemical system are essentially determined by intermolecular reactions, and a number of properties must be considered when determining this relationship. Molecular shape and conformation, electrostatic, dipolar, and multipolar interactions, and short-range dispersive interactions are among these influences.[2], [3] Fluorescent dyes have thus become common molecular probes for determining microenvironmental parameters such as media polarity, as well as tracking their distribution and relocation in micro heterogeneous systems.

Based on the literature study, it is reported that researchers have been interested in obtaining white light emission from organic molecules because of their usefulness in the solid-state lighting industry, as well as the light effects of liquid-crystal displays (LCDs) and full color OLED displays.[4], [5] Very rare organic compounds have been reported that emit across the entire visible spectrum (400 nm to 700 nm) with only a few reports available.[6]–[8] However, multilayered device fabrication by successive evaporation of different emitting compounds, spin coating of a mixture of different soluble emitters, excimer or exciplex emission, use of organic–metal complexes in a hybrid device structure, and phosphorescent emitters with a compatible host are the most commonly used methods for white light emissions.[4], [6], [9] [10], [11]. For white emission, the system structure is also essential. The

disadvantages of phosphorescent emitters are their high expense and complex system architectures. As a result, in order to be realistic in White organic light emitting diodes (WOLEDs), emission from a single compound is often preferred for ease of product fabrication.[9], [12]–[14] WOLEDs with a single emitting part provide benefits such as reproducibility, system reliability, and ease of fabrication[14]. In this context, the field of developing new fluorescent organic moieties with a broad spectrum of emissions in their solid state remains intriguing and demanding for researchers[15], [16]

Coumarins have inspired researchers' attention for decades due to their broad variety of uses in electronics and photonics, taking advantages of their high stability and ease of synthesis, which has led to many sophisticated applications as sensors and emitters for solid state dye lasers and OLEDs.[14], [17], [18] However, based on structures the planar coumarin are considered to be more prone to self-quenching due to aggregation and intermolecular interactions (n–n interactions), reducing their usage and applications in OLEDs.[2], [19]–[23] It is believed that the invention of a host–guest doped emitter device has been a blessing to emissive display technologies increasing its application in many fields.[5], [24]–[28] Simultaneously, a lot of work has been undertaken to eliminate planar luminophores' aggregation/dye–dye interactions. Regulated n–n interactions are important in the creation of long-range emissions (excimer/exciple formation), so they've been used as a support for light generation, especially white electroluminescence (EL).[28], [29] As a result, over the last decade, new fluorescent materials for OLEDs, as well as the creation of a basic device structure, have gotten a lot of attention.[6], [17], [30], [31] In this context, our exploration for new white-emitting electroluminescent materials led us to synthesize planar coumarin derivatives with extended n-conjugation. The physical properties in solution such as UV-vis absorption, fluorescence and excited-state lifetime of compound **1** are investigated in this study.

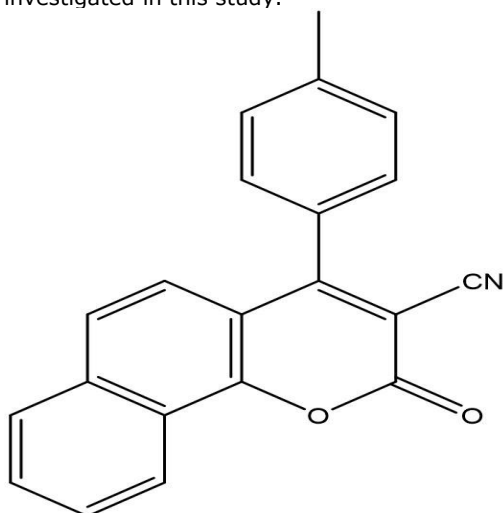


Figure 1. Chemical structures of **1**, see experimental section.

## Results and discussion

Table 1 summarizes the collected major absorption and emission maxima for chromophore **1**'s structures, as well as the single fluorescence lifetime values (Figures 2, 3), along

with the corresponding Stokes shift, relative fluorescence quantum yields ( $\Phi_F$ ), excited-state lifetime ( $\tau$ ), radiative ( $k_r$ ) and non-radiative ( $k_{nr}$ ) rate constants in different neat solvents.

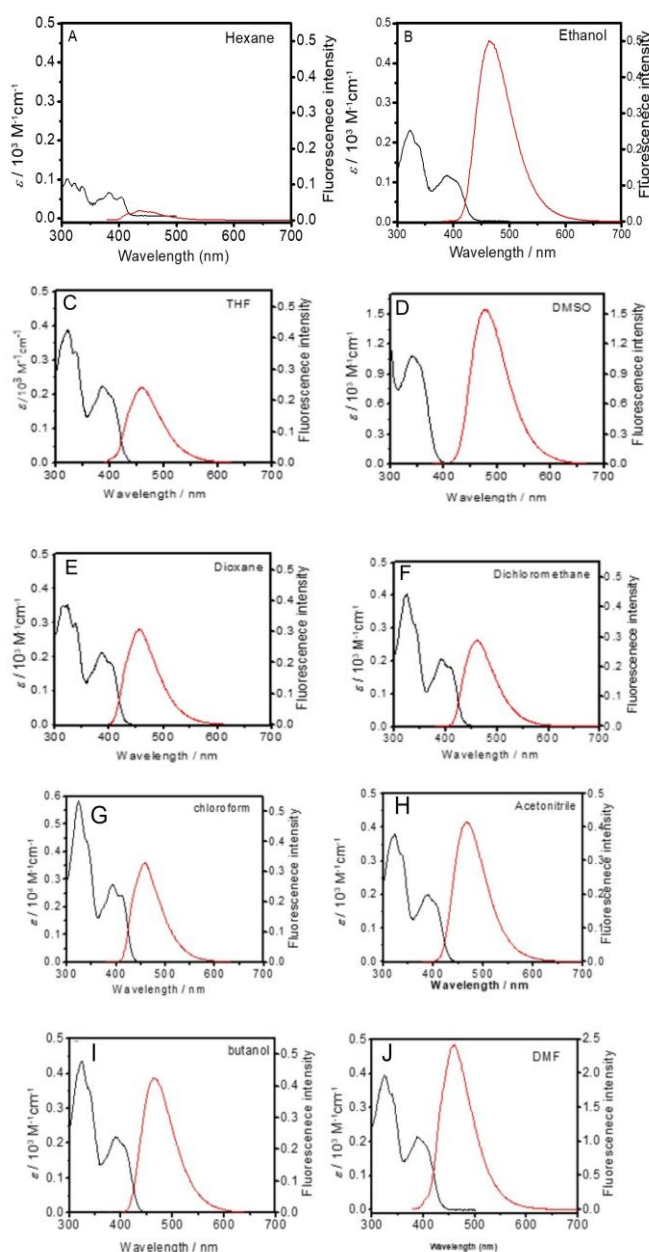
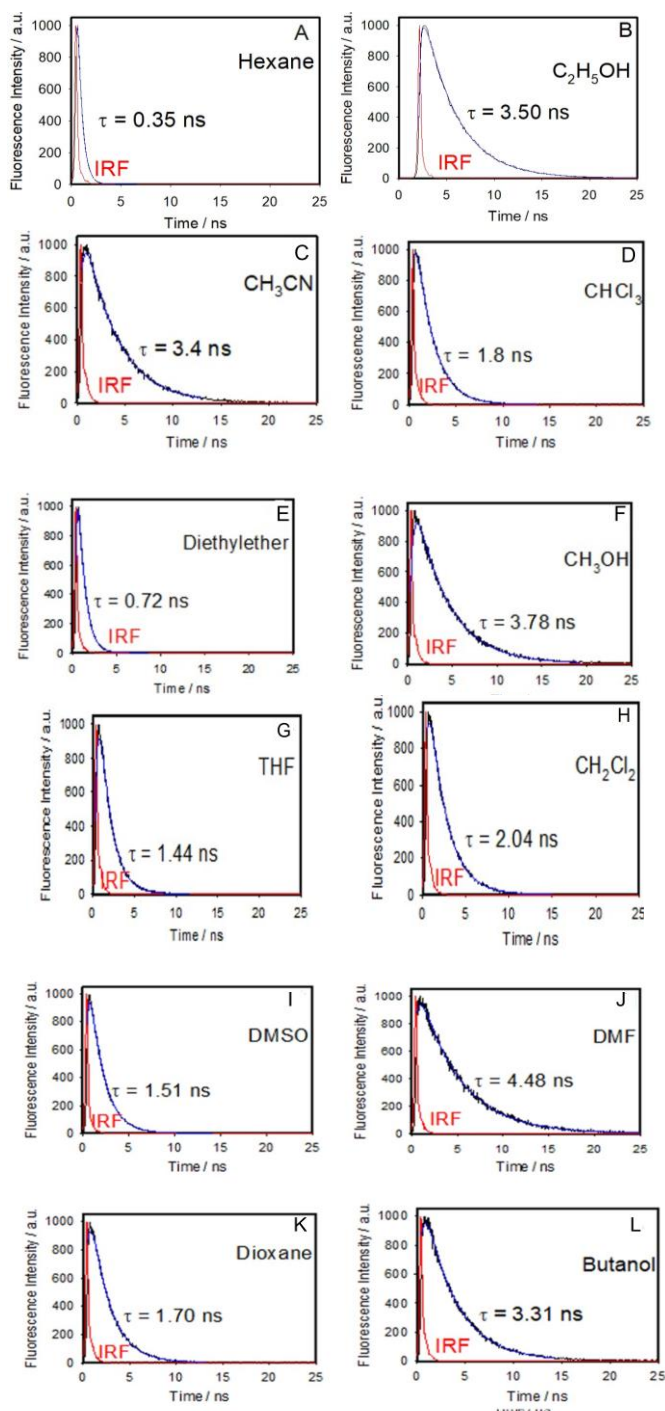


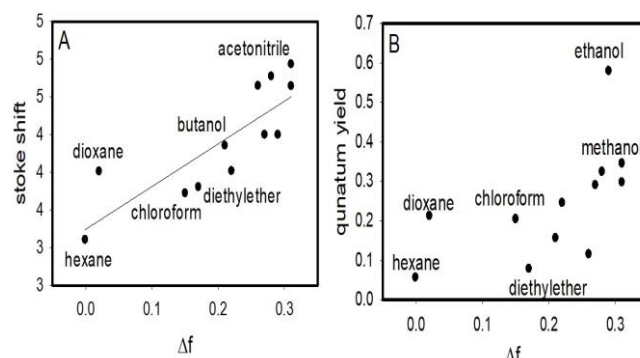
Figure 2. Absorption (black) and emission (red) spectra ( $\lambda_{\text{exc}} = 375 \text{ nm}$ ) of chromophore **1** in hexane (A) and ethanol (B), tetrahydrofurane (C), dimethylsulphoxide (D), dioxane (E), dichloromethane (F), chloroform (G), acetonitrile (H), butanol (I), dimethyleflouroamide (J)



**Figure 3.** Emission decays for **1** (25  $\mu$ M) in hexane (A), ethanol (B) acetonitrile (C), chloroform (D), diethylether (E), methanol (F), tetrahydrofuran (G), dichloromethane (H), dimethylsulphoxide (I), dimethylefouramide (J), dioxane K) and Butanol (L) upon excitation at 375 nm (30 ps) at room temperature. Instrument response function (IRF) is also shown in red upon excitation at 375 nm (30 ps) at room temperature. Instrument response function (IRF) is also shown in red.

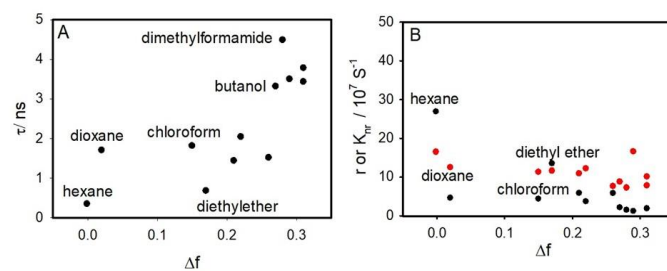
Noticeably, non-linear Lippert-Metaga plot has been observed as illustrated in Figure 4A when stokes shift values were plotted against solvent orientational polarizability ( $\Delta f$ )

(Table 2). In addition, the measured  $\Phi_F$  and  $\tau$  did not correlated with solvent polarity/polarizability factors (Figure 4B and 5A). This means that solvent polarity and polarizability effects do not alone play a role on affecting the photophysical properties of **1** in neat solvents.



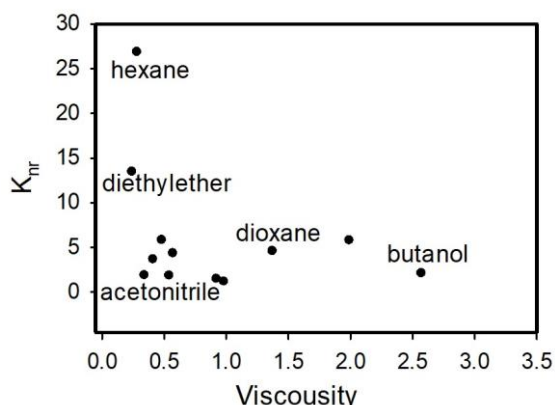
**Figure 4.** Correlation between the stokes shifts ( $\Delta\nu$ , A) and fluorescence quantum yields ( $\Phi_F$ , B) for **1** with the orientation polarizability ( $\Delta f$ ) for different solvents.

The time-resolved emission measurements along with the quantum yields data enabled us to better understand the origin of luminescence and to assess which solvent property controls the spectral properties of **1**. In the present study, the emission decays were measured every 10 nm across the entire emission spectra for each sample when excited at 375 nm. In the global analysis, the data measured at all wavelengths are fitted simultaneously by a mono-exponential function convoluted with the instrument response function (IRF). The fluorescence lifetime is given by the inverse of the rate constant of the exponential decay. The fluorescence lifetime experiments were repeated several times. The estimated experimental error was 4 %. The radiative ( $k_r$ ) and non-radiative ( $k_{nr}$ ) rate constants (Table 1) were then calculated and compared to the solvent parameters listed in Table 2 (Figure 5B). While  $k_r$  is independent of solvents polarity, value of  $k_{nr}$  increases significantly in low-polarity solvents.



**Figure 5.** Correlation between the excited-state lifetime ( $\tau$ , A), radiative ( $k_r$ ) and non-radiative ( $k_{nr}$ , B) decay rates for **1** with the orientation polarizability ( $\Delta f$ ) for different solvents.

To explain the above observation, the  $k_{nr}$  was plotted against solvent viscosity. An exponential dependence was observed (Figure 6). It transpires that by analogy to similar coumarin derivatives,<sup>2</sup> the increased value of  $k_{nr}$  in non-polar solvents is attributed to the rotation of toluene or cyano group, which provides a rapid deactivation pathway to the ground state. In more polar solvents an intramolecular charge state (ICT) state is formed, causing the larger Stokes shift. However, in this ICT state, rotation of the toluene is restricted and  $k_{nr}$  decreased. It must be said that this rotation is restricted due to large energy barrier in the ground state. The energy barrier is much smaller in the excited state. Consequently, such rotation affects the emission intensity and excited state lifetime of the fluorophore.



**Figure 6. Exponential dependence of non-radiative decay rates for 1 with solvent viscosity ( $\eta$ ) in cP for different solvents,  $R = 0.99$**

## Experimental

### Samples

All solvents CB7 (purity >99.9%) were purchased from Sigma-Aldrich and used as received. Millipore water was used (conductivity less than 0.05  $\mu$ S).

### Spectroscopy

The UV-visible absorption spectra were measured between 300 and 500 nm on a Cary-300 instrument (Agilent, Santa Clara, CA, USA) at room temperature. Fluorescence spectra measurements were scanned at room temperature, between 400 and 700 nm on a Cary-Eclipse fluorimeter (Agilent, Santa Clara, CA, USA). Slit widths were 2.5 nm for both excitation and emission monochromators. The pH values were recorded using a pH meter (WTW 330i equipped with a WTW SenTix Mic (Xylem Analytics Germany Sales GmbH & Co. KG, WTW, Weilheim, Germany). Quartz cuvettes (1.0 cm, 4.0 mL) were used in all spectroscopic measurements and were obtained from Starna Cells Inc. (Atascadero, CA, USA). The concentration of chromophore was kept at 25  $\mu$ M in all experiments, in which solution was prepared under  $N_2$  purging. All NMR spectra were performed on a Varian 400 MHz spectrometer in  $D_2O$ . All  $^1H$ -NMR spectra are referenced in ppm with respect to a TMS standard. The pH values of the solutions were adjusted ( $\pm 0.2$  units) by adding adequate amounts of HCl or NaOH and recorded using a pH meter (WTW 330i equipped with a WTW SenTix Mic glass electrode).

### Photochemistry

The excited-state lifetimes were measured by time-correlated single-photon counting (TCSPC) on LifeSpec II spectrometer (Edinburgh, Kirkton Campus, UK) by using EPL-375 picosecond diode laser ( $\lambda_{ex} = 375$  nm, repetition rate = 20 MHz, and instrument function = 30 ps) for excitation. A red-sensitive high-speed PMT detector (H5773-04, Hamamatsu Photonics K. K., Hamamatsu, Japan) was used to collect  $\sim 10,00$  counts/s for each run. The concentration of **1** was 25  $\mu$ M in each solvent. The data were analyzed by the iterative reconvolution method using the instrument's

software that utilizes the Marquardt-Levenberg algorithm to minimize  $\chi^2$ . The fluorescence decay in each solvent was found to fit mono-exponential function, eq. (1),

$$I(t) = a \exp(-t/\tau) \quad (1)$$

where  $\tau$  is the lifetime with amplitude  $a$

### Quantum Yield, Radiative and Non-Radiative Measurements

$\Phi$  values were measured using Coumarin 2 (C 450) in acetonitrile as the standard ( $\Phi_F = 0.8$ ),<sup>2</sup> and calculated using the known equation:

$$\Phi_{unk} = \Phi_{std} \left( \frac{I_{unk}}{A_{unk}} \right) \left( \frac{A_{std}}{I_{std}} \right) \left( \frac{n_{unk}}{n_{std}} \right)^2$$

where  $n$  is the refractive indices for the standard (std) and experimental (unk) solvents,  $I$  is the fluorescence integral of the emission between 400 and 700 nm, and  $A$  is the absorbance at the excitation wavelength. The error estimated as standard deviation of the mean was approximately 10% for the fluorescence quantum yields.  $k_r$  and  $k_{nr}$  were calculated using the known equations:

$$\Phi = \frac{k_r}{k_r + k_{nr}}, \tau = \frac{1}{k_r + k_{nr}}, k_{nr} = \frac{1 - \Phi}{\tau}, \text{ and } k_r = \frac{\Phi}{\tau}$$

## Conclusions

The detailed photophysics of a coumarin-based dye as a function of local microenvironment is described and it is concluded that the change in quantum yields and excited-state lifetimes are due to the changes in non-radiative rate constants ( $k_{nr}$ ), which correlates exponentially with solvent viscosity. A more polar solvents (such as butanol) restricts the rotation of the toluene/cyano compared to non-polar solvent (such as hexane).

## Acknowledgements

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**Table 1.** Absorption ( $\lambda_a$ ), steady-state emission ( $\lambda_f$ ) peak positions and excited-state lifetimes  $\tau$  along with stokes shifts ( $\Delta\nu$ ), fluorescence quantum yields ( $\Phi_F$ ), radiative rate constants ( $k_r$ ), and non-radiative rate constants ( $k_{nr}$ ) for **1** at 25  $\mu\text{M}$  in neat solvents. Dye was excited at 375 nm in all solvents.

Solvents	$\lambda_a/\text{nm}$	$\lambda_f/\text{nm}$	$\Delta\nu(\text{cm}^{-1})$	$\Phi_F$	$\tau/\text{ns}$	$k_r/10^8\text{ s}^{-1}$	$k_{nr}/10^8\text{ s}^{-1}$
hexane	383	437	3226.4	0.057	0.35	16.5	26.9
diethylether	387	452	3715.9	0.079	0.682	11.6	13.5
chloroform	393	459	3658.8	0.205	1.817	11.3	4.37
dichloromethane	392	462	3865.2	0.246	2.044	12.2	3.69
dioxane	387	455	3861.8	0.213	1.708	12.5	4.61
tetrahydrofuran	387	460	4100.7	0.157	1.441	10.9	5.85
acetonitrile	382	469	4856.1	0.346	3.435	10.1	1.9
methanol	387	472	4653.4	0.298	3.782	7.8	1.86
ethanol	389	465	4201.6	0.580	3.501	16.6	1.2
dimethylformamide	387	474	4742.7	0.325	4.489	7.23	1.5
butanol	389	465	4201.6	0.291	3.319	8.78	2.13
dimethylsulphoxide	391	478	4654.9	0.116	1.518	7.64	5.82

**Table 2.** Solvent properties including refractive index ( $n$ ), dielectric constant ( $\epsilon$ ), orientational polarizability ( $\Delta f$ ), viscosity ( $\eta$ ), hydrogen bond accepting ability ( $\beta$ ), hydrogen bond donating ability ( $\alpha$ ) and polarity/polarizability parameter ( $\pi^*$ ).

Solvents	N	$\epsilon$	$\Delta f$	$\eta/\text{cP}$	$\alpha$	$\beta$	$\pi^*$	$\alpha + \beta + \pi^*$
hexane	1.376	1.87	-0.001	0.28	0	0	-0.08	-0.08
diethylether	1.352	4.33	0.17	0.24	0	0.47	0.27	0.74
chloroform	1.446	4.81	0.15	0.57	0.44	0	0.58	1.02
dichloromethane	1.416	8.93	0.22	0.41	0.33	0	0.82	1.15
dioxane	1.422	2.22	0.02	1.37	0	0.37	0.55	0.92
tetrahydrofuran	1.406	7.58	0.21	0.48	0	0.55	0.58	1.13
acetonitrile	1.344	37.5	0.31	0.34	0.19	0.31	0.75	1.25
methanol	1.328	32.7	0.31	0.54	0.93	0.62	0.6	2.15
ethanol	1.364	25.07	0.29	0.98	0.83	0.77	0.54	2.14
dimethylformamide	1.435	37.6	0.28	0.92	0	0.69	0.88	1.57
butanol	1.394	17.85	0.27	2.57	0.79	0.88	0.47	2.14
dimethylsulphoxide	1.479	48.9	0.26	1.99	0	0.76	1	1.76

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