RGB Emission through Controlled Donor Self-Assembly and Modulation of Excitation Energy Transfer: A Novel Strategy to White-Light-Emitting Organogels

By *Chakkooth Vijayakumar, Vakayil K. Praveen,* and
*Ayyappanpillai Ajayaghosh**

White-light-emitting organic materials have attracted much attention because of their potential applications in display and lighting devices.[1] An ideal white-light-emitting system needs to emit the three primary RGB (red, green, and blue) colors in required intensities that cover the visible wavelength range from 400 to 700 nm. One of the most exploited strategies for this purpose is the use of physically blended multicomponent systems that simultaneously emit at the RGB wavelength region. In many cases, partial energy transfer between donor–acceptor molecules has been exploited for the generation of white light. Different types of materials based on π-conjugated polymers, metal complexes, and low-molecular-weight organic molecules have been used for white-light emission, either in solution or in the solid state.[2–4] However, to date, a white-light-emitting organogel has not been reported, although organogelators with tunable emission properties are known.[5–7] Chromophore-based self-assemblies and gels are efficient scaffolds for the design of supramolecular light,harvesting assemblies.[8,9] As a result of efficient excitation-energy migration, energy transfer in oligo(*p*-phenylenevinylene)s (OPV) self-assemblies and gels occurs even to distantly located acceptors, present in small quantities through funnelling of excitation energy.[10,11] Herein, we report a rational strategy to the design of a hitherto unknown white-light-emitting organogel using a novel concept of functional-group-controlled donor self-assembly and consequent modulation of excited-state properties in the supramolecular gel state.

The motivation for the present study stems from our recent observation of a temperature-dependent emission color change from a red gel to a blue solution through the formation of an intermediate white-light-emitting solution.[11d] However, we were not able to obtain a white-light-emitting gel in this case, since, in the gel state, the emission changes to red. Therefore, we took it as a challenge to design a white-light-emitting organogel that is successfully materialized and reported in the present study. From our experience, it is clear that control of the self-organization of the donor is crucial for energy migration, which in turn will regulate the energy-transfer processes either partially or completely, leading to tunable emission colors. In the event of a moderate self-assembly and partial energy transfer of a blue-light-emitting donor to a red-light-emitting acceptor, a mixture of blue, green, and red emissions could be simultaneously generated, leading to a white-light-emitting gel. On the other hand, in the case of a strong self-assembly and complete energy transfer, only red emission of the acceptor will occur. As a proof-of-principle of this hypothesis, we illustrate controlled energy transfer in bis- and monocholesterol-appended OPV derivatives, **1** and **2**, in the presence of an acceptor **3**, to result in white and red emissions, respectively (see Fig. 1). Moreover, we illustrate here how important functional groups are in regulating the self-assembly and electronic properties of chromophores to achieve white-light-emission exclusively in a gel state.

We have already shown that the bischolesterol–OPV (**1**) forms a weak gel (critical gelator concentration (CGC) = 7 mg mL^{-1} in decane) through the hierarchical self-assembly of pseudo-H-type aggregates that lead to twisted helical tapes, whereas the monocholesterol–OPV (**2**) forms a strong gel (CGC = 3.4 mg mL^{-1} in decane) comprised of coiled helical tapes in which the chromophores are packed as pseudo-J-type aggregates.[12] This difference in the molecular packing results in distinct optical, chiroptical, and morphological properties. In light of these observations, we speculated that the excited-state dipole orientation of **1** and **2** in the twisted and coiled helical states, respectively, may be considerably different, and may strongly influence the excited-state properties, particularly the excitation energy migration. This is established by time-resolved emission studies (TRES) of **1** and **2** (3×10^{-4} M) in the gel state in decane at different time settings after excitation at 375 nm (Fig. 2). In both cases, a red-shift was observed in the emission spectrum with an increase in time. This dynamic red-shift of the emission maximum with time is a result of the increased population of the singlet excited states of the higher-order aggregates with a low HOMO–LUMO energy gap through excitation energy migration.[13,14] Interestingly, in the case of **1**, the red-shift of the emission maximum (494 nm) was marginal, although the shoulder band at 528 nm intensified with time (Fig. 2a). The spectrum obtained after 1.46 ns was almost identical to the steady-state emission of the decane gel of **1**. However, in the case of **2**, with an increase in time after excitation, the emission spectrum becomes broad with a significant red-shift in the emission maximum (Fig. 2b). Moreover, within 672 ps after excitation, the emission spectrum resembles the steady-state emission. These observations reveal a slower excitation energy migration in **1** when compared with that of **2** in the gel state. It

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[*] Dr. A. Ajayaghosh, Dr. C. Vijayakumar, Dr. V. K. Praveen
Photosciences and Photonics Group
Chemical Sciences and Technology Division
National Institute for Interdisciplinary Science and
Technology (NIIST)
CSIR, Trivandrum 695 019 (India)
E-mail: ajayaghosh62@gmail.com

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must be mentioned that both **1** and **2** did not show any change in TRES in the solution state.

The wavelength dependence of the emission decay of **1** and **2** in the gel state in decane provided strong evidence for the difference in the rate of energy migration (Fig. 3). The lifetime-decay profiles collected at lower wavelengths indicate a fast process when compared to that at higher wavelengths. In both cases, the decay monitored at higher wavelengths exhibited a growth component at shorter time scales, indicating that the population of the singlet excited states of the higher-order aggregates occurs through energy migration from lower-order aggregates.[14] The growth components of **1** and **2** correspond to 764 and 332 ps, respectively, indicating that the excitation energy migration is slow in the former when compared with that of the latter. Additional proof for the difference in energy-migration efficiencies in these two systems was obtained from fluorescence anisotropy decay studies.[15]

From the above observations, we anticipated that, because of the weak gelation and slow exciton diffusion, partial energy transfer may occur from the aggregates of **1** to a red-light-emitting acceptor, which leads to a mixture of the RGB emission. On the other hand, the tendency for strong gelation and fast exciton diffusion may facilitate efficient energy transfer from **2**, which leads to exclusive red emission. In order to establish this hypothesis, we have chosen a red-light-emitting conjugated oligomer **3** as the acceptor.[16] Energy-transfer studies were carried out by mixing appropriate volumes of the acceptor from a stock solution to a decane solution of the donor. The concentration of the donor is maintained at 3×10^{-4} M while the amount of acceptor varied from 0 to 2 mol%. The mixture was then heated to 80 °C to form a homogeneous solution, and slowly cooled to room temperature to form coassembled gels. The CGC of the donor and the acceptor mixture (**1** and **3** or **2** and **3**) in decane was found to be slightly less when compared with that of the corresponding donors, which indicates the encapsulation of the acceptor with the donor assembly.[15] The energy transfer was monitored in the gel state by recording the emission of OPVs in the absence and presence of acceptor after excitation of OPVs at 380 nm. Excitation of the decane gel of **2** with the addition of small quantities of **3** (0–2 mol%) resulted in gradual quenching of the broad emission of the former (Fig. 4a). Simultaneously, a relatively sharp emission at 613 nm, which corresponds to the red emission of the

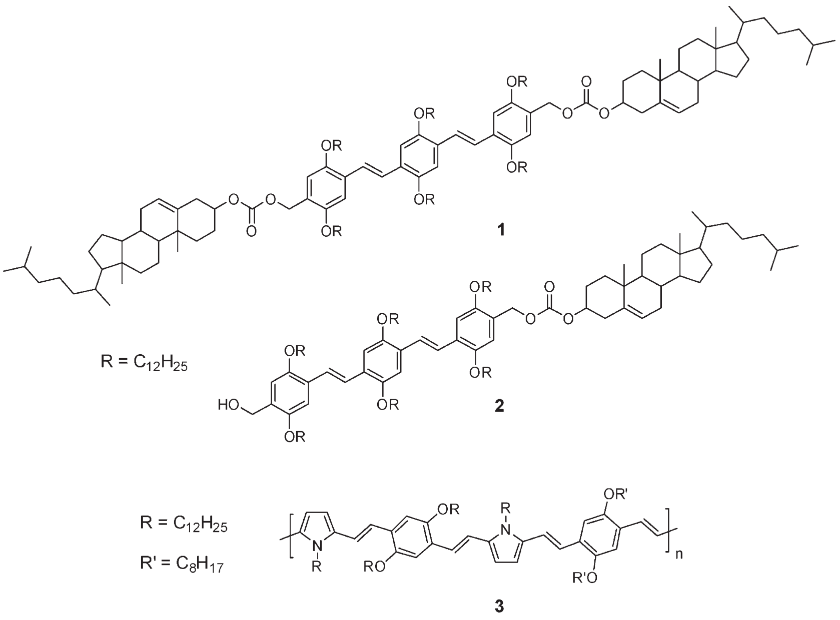

Figure 1. Structure of molecules **1**–**3**.

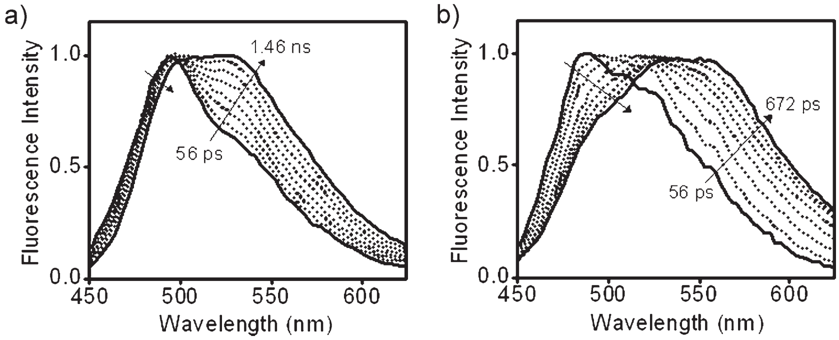

Figure 2. Normalized time-resolved emission spectra of a) **1** and b) **2** in decane at room temperature ($c = 3 \times 10^{-4}$ M, $l = 1$ mm, and $\lambda_{ex} = 375$ nm), indicating the difference in the excitation energy migration.

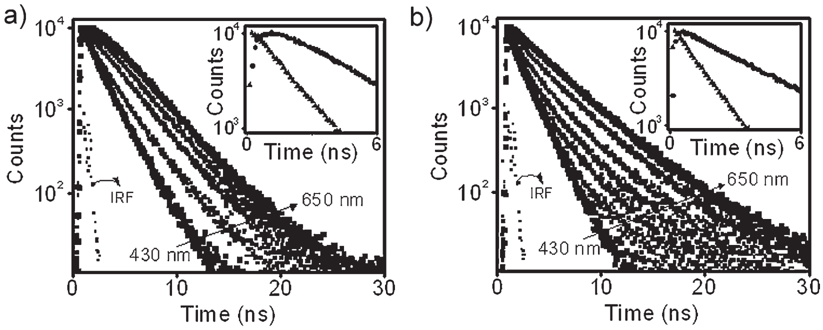

Figure 3. Wavelength-dependent fluorescence decay of a) **1** and b) **2** in decane at room temperature ($c = 3 \times 10^{-4}$ M, $l = 1$ mm, and $\lambda_{ex} = 375$ nm). Inset shows the lifetime-decay profiles monitored at 430 nm (▲) and at 650 nm (●), showing the growth at the initial time scale. IRF = instrument response function.

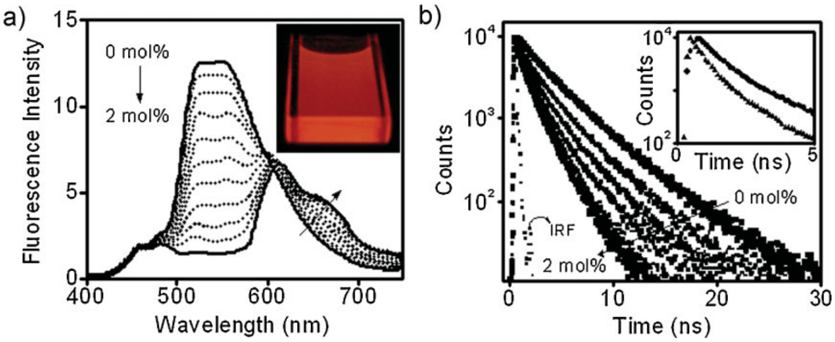

Figure 4. Changes in the a) fluorescence emission ($\lambda_{ex} = 380$ nm) and b) lifetime-decay profiles ($\lambda_{ex} = 375$ nm, monitored at 560 nm) of **2** on addition of increasing amounts of **3** (0–2 mol%) in decane at room temperature ($[\mathbf{2}] = 3 \times 10^{-4}$ M, $l = 1$ mm). Insets show a) the red-light emission of the gel on irradiation using 365 nm light and b) fluorescence-decay profiles of **3** in the absence (▲) and presence (●) of **2**.

acceptor, was formed with a maximum of 90% quenching of the donor emission. No spectroscopic changes were observed on addition of more than 2 mol% of the acceptor. However, a large excess of acceptor destabilizes the gel assembly, leading to a decrease in the energy-transfer efficiency. The fluorescence-lifetime-decay profiles of **2** in decane gel ($\lambda_{ex} = 375$ nm) exhibited biexponential decay with lifetimes of 2.47 ns (54.4%) and 4.82 ns (45.6%) in the absence of the acceptor when monitored at 560 nm ($\chi^2 = 1.12$, Fig. 4b). In presence of 2 mol% of the acceptor, **2** exhibited fast biexponential decay with time constants of 0.58 ns (73.3%) and 1.8 ns (26.7%, $\chi^2 = 1.07$). Such acceleration in decay dynamics of the donor in the presence of the acceptor provides clear evidence of nonradiative energy transfer, and rules out the possibility of a trivial radiative mechanism.[13c–13e] This was further proved from the difference observed for the decay of **3** in the absence and presence of **2** (inset, Fig. 4b). The growth component at the initial time scale represents the population build up of the excited state of **3** through nonradiative energy transfer from **2**.[13e,17]

Interestingly, the emission of a decane gel of **1** upon excitation at 380 nm in the absence of **3** showed two maxima, at 492 and 528 nm, with shoulders on either side (Fig. 5a). The shoulder at 464 nm (blue-light-emission color) corresponds to the residual monomer emission of the donor. Upon excitation at 380 nm in the presence of small amounts of **3**, the emission was quenched with the concomitant formation of a new peak at 607 nm, which corresponds to the acceptor emission, without affecting the blue-light-emitting shoulder at 464 nm. In this case, addition of up to 2 mol% of **3** could cause only nearly 63% quenching (when monitored at 528 nm) of the emission, in contrast to the 90% quenching in the case of **2**. It must be noted that a further increase in the amount of **3** while maintaining the same donor concentration dissociates the donor gel scaffold, leading to a significant decrease in the energy-transfer efficiency. The fluorescence-lifetime-decay profiles (Fig. 5b, $\lambda_{ex} = 375$ nm) of **1** in the gel state when monitored at the aggregate emission exhibited a biexponential decay ($\chi^2 = 1.17$) with lifetimes of 2.38 ns (79.6%) and 3.79 ns (20.4%). In the presence of 2 mol% of **3**, the decay ($\chi^2 = 1.18$) became fast, with time constants of 1.85 and 0.57 ns and relative amplitudes of 93% and 7%, respectively. Noticeably, the fluorescence decay of **3** in the presence of **1** was found to be slow, with a profound growth component at the initial time scale (Fig. 5b, inset) when compared with that of **3** in the presence of **2** (Fig. 4b, inset).

Surprisingly, when the concentration of the acceptor reached around 2.0 mol%, the decane gel of **1** exhibited a near-white-light emission, with Commission Internationale de L'eclairage (CIE) coordinates of (0.28,0.34). The partial quenching of the emission of the donor self-assembly results in a broad emission spectrum in the range of 400–750 nm, which contains red, green, and blue colors that yield a white-light emission. From this observation, it is clear that the blue emission of the donor monomers, green emission of the donor aggregates, and red emission from the acceptor as a result of partial energy transfer occur simultaneously. The purity of the white-light emission could be improved by optimizing the concentration of **1** (3.2×10^{-4} M) and **3** (2.1 mol%). At this stage, the emission spectrum covers the visible-light range (Fig. 6a) with a better balance of red, green, and blue, which results in a pure white-light-emitting gel (Fig. 6a, inset). The chromaticity diagram at this particular composition reveals CIE coordinates (0.31,0.35) (Fig. 6b), which are ideal for white-light emission according to the 1931 CIE coordinate diagram.[18]

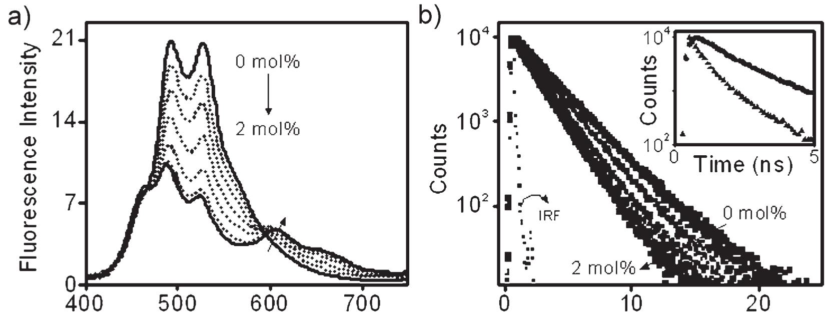

Figure 5. Changes in the a) fluorescence emission ($\lambda_{ex} = 380$ nm) and b) lifetime-decay profiles ($\lambda_{ex} = 375$ nm, monitored at 528 nm) of **1** on addition of increasing amounts of **3** (0–2 mol%) in decane at room temperature, indicating partial energy transfer that results in a broad emission ($[\mathbf{1}] = 3 \times 10^{-4}$ M, $l = 1$ mm). Inset shows the fluorescence-decay profiles of **3** in the absence (▲) and presence of **1** (●).

In summary, we have succeeded in the rational design of a hitherto unknown white-light-emitting organogel, and proved that excited-state properties and energy transfer in a donor self-assembly could be controlled using the principles of supramolecular chemistry. Moreover, this study highlights that even electronically insulated functional groups may help to modulate the optoelectronic properties of a given chromophore through controlled self-assembly. Even though, at this point, we are not sure of a practical application for the white-light-emitting gel, it could be a potential

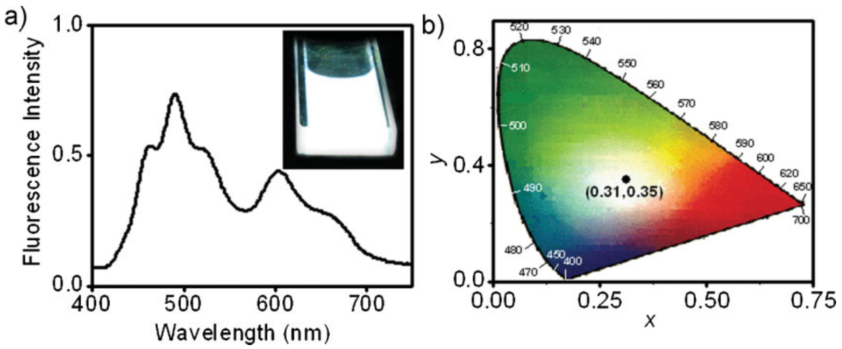

Figure 6. a) Fluorescence emission spectrum of a decane gel of **1** (3.2×10^{-4} M) in presence of 2.1 mol% of **3** ($\lambda_{ex} = 380$ nm, $l = 1$ mm). Inset shows the white-light emission of the gel on irradiation using 365 nm light. b) The corresponding 1931 CIE coordinate diagram of the white light emission ($x = 0.31$, $y = 0.35$).

candidate in future technologies, and hence may generate considerable research interest.

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