Organic composite materials: Understanding and manipulating excited states toward higher light-emitting performance

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Abstract
Organic composite materials have been attracting extensive research interest for light-emitting applications. A wide variety of luminescent organic composite materials have been synthesized, which are of great significance for both the investigation of basic photophysics and the realization of high-performance photonics devices. Function-oriented syntheses of luminescent organic composite materials rely on the understanding and manipulating of molecular excited states. In this review, we focus on the discussion about the structure design and dynamics modulation of the electronic excited states in the organic composite materials. The excited-state structures and dynamics involve singlet/triplet levels, vibronic transition, charge transfer, and energy transfer, and so on, while the light-emitting behaviors include fluorescence, phosphorescence, persistent luminescence, electroluminescence, and lasing. We aim to give insight into the relationship between light-emitting properties and excited states of organic composite materials, which is beneficial for reaching higher tiers of design and applications of luminescent organic composite materials.

KEYWORDS
excited states, molecular optoelectronics, organic composite materials, organic lasers, organic light-emitting materials

1 | INTRODUCTION

Luminescence is an old subject, going back to the 19th century.⁴¹ Nowadays, diverse luminescent materials are ubiquitous in every section of modern society, such as lighting, display, scintillation, and anti-counterfeiting. Among all luminescent materials, organic compounds exhibit many superiorities, including low-cost processing, large active area, mechanical flexibility, and color tunability.⁵² An early observation of fluorescence from the organic material (i.e., malaline) was described in 1560. However, it was not until 1966, when the first dye laser was demonstrated,⁶¹ that the luminescent organic molecules started to receive much attention. Organic dyes are suitable for tunable and pulsed lasers, finding many applications in astronomy, manufacturing, medicine, and spectroscopy.⁴⁵ In 1987, the first efficient organic light-emitting diode (OLED) was built.⁶ With the commercialization of OLED display panels, the organic photonics industry has come of age.⁷¹ Overall, the development and innovative use of new luminescent organic materials continually generate new knowledge and applications.⁸–¹³

It is well established that the light-emitting properties of organic materials are mainly governed by their electronic excited states, which are in turn determined by their molecular and supramolecular structures.¹⁴–¹⁵ To obtain new luminous organic materials with higher performances or novel functionalities, several strategies have been developed that make use of molecule design, packing mode control, and component combination to manipulate the electronic excited states. The molecular design is the most direct and effective strategy to obtain new luminescent organic compounds¹⁴ but requires difficult and time-consuming synthetic processes. The packing mode control via self-assembly induces the formation of different luminescent aggregates from the same organic compounds¹⁶–¹⁹ but suffers from limited applicability. The component combination provides an effective and universal platform to synthesize the organic materials and architectures with novel light-emitting properties. More interestingly, the composite materials not only inherit the advantages of each component but also produce additional functionalities through the synergetic matter/energy interactions between multiple components.²¹–²²
So far, a wide variety of organic composite materials have been developed for light-emitting applications. The component combination effectively modulates the structure and dynamics of the electronic excited states, resulting in lots of novel luminescent phenomena and applications. Several reviews have summarized this fast-growing field but mainly focused on discussions about material synthesis for specific topics, such as fluorescence, phosphorescence, lasing, and photofunctional devices. This review highlights the understanding and manipulating of excited states in luminescent organic composite materials. We start with a brief introduction to composite material chemistry, including materials syntheses and structures. Following this is a comprehensive discussion of composite material photophysics, mainly concerning the structure design and dynamics modulation of the electronic excited states. The excited-state structures and dynamics involve singlet/triplet levels, vibronic transition, charge transfer, and energy transfer, and so on, while the light-emitting behaviors include fluorescence, phosphorescence, persistent luminescence, and lasing. Special emphasis is put on gaining insight into the relationship between light-emitting properties and excited states of organic composite materials, and therefore providing guidance for the photofunction-oriented organic composite material design. Finally, we give a conclusion as well as a prospect of future research directions in this promising field.

2 | COMPOSITE MATERIAL CHEMISTRY

Organic compounds provide an ideal platform for the exploration of luminescent composite materials due to their unique characteristics of good compatibility, high excited-state flexibility, and molecular diversity. High material compatibility is a prerequisite for the combination of multiple components. The highly flexible excited-state structures and dynamics benefit the realization of novel light-emitting properties. The molecular diversity enables an almost infinite pool of synthetic components toward a wide variety of luminescent composite materials. A central goal of developing luminescent composite materials is to obtain novel light-emitting properties beyond the individual components. Since the luminescence properties highly depend on the composite material compositions and structures, considerable research effort has been dedicated to developing various rational construction strategies of luminescent organic composite materials, such as cooperative assembly, epitaxial growth, templated combination, and post-transfer integration.

In organic composite materials, different molecular components are held together by weak non-covalent interactions (Figure 1A), including van der Waals force, π−π stacking, hydrogen bond, halogen bond, and charge transfer interaction. According to composition distribution, the organic composite materials can be classified into three categories, supramolecular, doped, and epitaxial structures (Figure 1B). From the viewpoint of luminescent distribution, the organic composite materials can be divided into two types, light-emitting homostructures and heterostructures. In the supramolecular structures, the molecular components have a certain stoichiometric ratio due to relatively strong inter-component interactions, such as host-guest inclusion, charge-transfer complexation, and halogen bonding. As a result of uniform composition distribution, the supramolecular structures exhibit uniform light emission. In the doped structures, dopants can be either uniformly or nonuniformly distributed, leading to uniform and nonuniform luminescence, respectively.

In the epitaxial structures, two material components interact only at the interfaces, which are more suitable for constructing photonic devices, such as tunable waveguides, optical routers, modulators, and sensors. We do not go into details of the epitaxial structures in this review article because they are summarized in many published reviews. In both supramolecular and doped structures, different material components interact with each other across the whole composite materials, resulting in a more significant influence on excited states. Hence, we discuss more supramolecular and doped structures to demonstrate the relationship between light-emitting properties and excited states of organic composite materials.
Organic composite materials control luminescence characteristics by reorganizing the excited-state structures (energy level height and potential barrier) and modulating the excited-state dynamics (population distribution and transition path/rate). The excited-state structures and dynamics of organic molecules can be well illustrated in a Jablonski diagram (Figure 2A). Upon absorbing a photon, the organic molecule undergoes a transition from the electronic ground state ($S_0$) to a high vibrational level of the first singlet excited state ($S_1$). Following this is a fast vibrational relaxation from a high vibrational level to the bottom of the first singlet excited state. For the organic molecules with large spin-orbit coupling, the first singlet excited state transfers to the first triplet excited state ($T_1$) through an intersystem crossing (ISC), which is also followed by a fast vibrational relaxation. For highly polarized organic molecules, the initially formed electronic excited state is also known as the localized excited (LE) state, which can further relax to an intramolecular charge-transfer (ICT) state. All these kinds of excited states finally decay back to the ground state via radiative and nonradiative transitions. Nonradiative transitions include internal conversions to the ground state via vibrational coupling and relaxation to other dark states (e.g., H-aggregate, excimer, and defect). Spontaneous emissions from the singlet and triplet excited states are called fluorescence and phosphorescence, respectively. Besides, the organic molecule offers a quasi-four-level system for laser generation. Stimulated emission transition occurs from the lowest singlet excited state to a vibrational level of the ground state.

Intermolecular energy transfer phenomena are ubiquitous in organic composite materials composed of multiple chromophores. The energy transfer behaviors via Coulombic interaction and electron exchange are known as Förster resonant energy transfer (FRET) and Dexter energy transfer (Figure 2B). The Förster mechanism is generally from a singlet donor to a singlet acceptor, while the energy transfer involving triplet states is often described by the Dexter mechanism.

### 3.1 Suppression of nonradiative transitions for efficient fluorescence/lasing

High radiative quantum yield is a prerequisite for luminescent materials to implement high-performance photonic devices. However, lots of important organic luminophores experience rapid nonradiative transitions due to intense intermolecular interactions in solid state, which is referred to as aggregation-caused quenching. Even for those organic luminophores with high radiative quantum yields in solid state, the exciton–exciton annihilation process at high excitation densities quenches the excited-state population. Spatially, separating the organic molecules by incorporating them as guests into host materials is an effective strategy to circumvent these intermolecular quenching effects. Transparent polymers are the most commonly used host materials, especially for versatile solid-state dye lasers but suffer from low loading concentrations of the luminescent molecules. In recent years, porous materials have emerged as excellent hosts allowing for high molecule loading concentrations. Metal-organic frameworks (MOFs) are particularly appealing as...
porous hosts because they provide spatial confinement for the guest organic molecules,[8] inducing unique (e.g., anisotropic and tunable) light-emitting properties.

Organic luminophores can be introduced into the highly regular channels/pores of MOFs via molecule diffusion or in situ self-assembly strategies. As a result, the organic luminophores experienced a huge luminescence enhancement. As shown in Figure 3, the 4-[4-[4-(dimethylamino)phenyl]-1,3-butadienyl]-1-ethyl quinolinium (LDS 798) exhibited very low fluorescence quantum yield (< 0.01 %) in solid-state owing to the formation of dark excimer states.[71] When incorporated into a zeolite-like MOF (rho-ZMOF), the fluorescence quantum yield of LDS 798 dramatically increased to 8.01% (Figure 3A–C). This indicated that the dispersion into the rho-ZMOF pores suppressed the intermolecular interactions between the dye molecules, avoiding the aggregation-caused quenching effect. The excitation density-dependent emission was investigated to further probe the exciton dynamics of LDS 798 in a rho-ZMOF microcrystal (LDS 798@MOF). The integrated photoluminescence (PL) intensity exhibited a clear linear relationship with the pump density as the nonlinear exponent $p = 1.01 \pm 0.02$ (Figure 3D), revealing that the exciton–exciton annihilation is completely suppressed in the LDS 798@MOF composite microcrystal. The absence of exciton–exciton interaction can be ascribed to the pore confinement effect of rho-ZMOF, spatially separating the LDS 798 molecules and minimizing the exciton–exciton interaction. Because of the suppression of undesired aggregation-caused quenching and exciton–exciton interaction, the LDS 798@MOF microcrystals supported efficient lasing (Figure 3E).[71]

3.2 | Enhancement of ISC for efficient phosphorescence

Phosphorescent organic materials are also highly attractive for their broad applications in OLEDs, bioimaging, and glow-in-the-dark.[26] So far, the most efficient phosphorescent chromophores are organometallic complexes suffering from high cost and toxicity.[79] Accordingly, the development of purely organic (metal-free) phosphorescent materials has become a research focus. Although several types of purely organic phosphorescent materials have been developed, low phosphorescence quantum yield is a formidable problem mainly due to low ISC yield and aggregation-induced quenching. The component combination provides an alternative avenue for realizing efficient phosphorescence from purely organic materials.

As shown in Figure 4A, 2,5-dihexyloxy-4-bromobenzaldehyde (Br6A) is a typical phosphorescence chromophore because lone-pair electrons on the aromatic carbonyl induce ISC via the spin-orbit coupling.[80] Nonemissive 2,5-dihexyloxy-1,4-dibromobenzene (Br6) crystal was selected as a host matrix to incorporate the Br6A. A similar molecular structure of the Br6 with Br6A facilitated the formation of high-quality mixed crystals. Strong halogen bonding (O⋅⋅⋅Br) exists between the Br6 and Br6A (Figure 4B), which significantly promotes the ISC (Figure 4C) because it induces the heavy-atom effect by delocalizing excited-state electrons from the Br6A oxygen to the Br6 bromine. Besides, the rigid crystal environment, together with the halogen bonding, effectively suppresses
the nonradiative internal conversion loss of the Br6A triplet states, while the molecular dispersion in crystal avoids the aggregation-induced quenching (Figure 4C). As a result, the Br6A doped in the Br6 crystals exhibited very strong phosphorescence with a quantum yield of up to 55% (Figure 4D,E). Such mixed crystal strategy is universal for organic conjugated compounds. Following the same design principle, three more sets of host-guest molecules were synthesized toward tunable phosphorescence (Figure 4F). A series of purely organic crystals with high phosphorescence efficiencies were synthesized (Figure 4G). With the electron density of the chromophore increasing, the emission colors of the mixed crystals changed from blue to red covering the whole visible light range (Figure 4G,H).

### 3.3 Storage of excited states for long persistent luminescence

Persistent luminescence is a phenomenon that a material stores excitation energy and maintains light emission for a long time, typically ranging from seconds to hours.\(^{[24,27,81]}\) Long persistent luminescence materials have been widely commercialized as glow-in-the-dark paints and are being explored for application in vivo biological imaging. Organic materials with persistent luminescence have recently captured much attention for their potential applications in large-area and flexible glow-in-the-dark paints, biomarkers, fabrics, and windows.

Typical long-lived emission from organic molecules is known as phosphorescence, which usually has a lifetime ranging from microseconds to milliseconds. A host-guest strategy was proposed to confine triplet excitons for persistent luminescence (Figure 5A).\(^{[82]}\) Deuterated organic compounds without heavy atoms were synthesized as phosphorescent guests. The exclusion of heavy atoms (e.g., metals and halogens) avoids the fast decay of triplet states induced by spin-orbit coupling. The deuteration of the phosphorescent guest reduces the nonradiative deactivation of triplet states.

Rigid steroidal compounds with higher triplet levels were chosen as hosts. The rigid host minimizes the triplet state quenching by the diffusional motion of the host. By confining the triplet excitons (Figure 5B,C), the hosts reduce both concentration and oxygen quenching.\(^{[83]}\) As a result,
the host-guest organic materials exhibit persistent phosphorescence combining high quantum yields (> 10%) and long lifetimes (> 1 s) (Figure 5A,D). Furthermore, such a host-guest material system allows for electrically excited persistent phosphorescence (Figure 5D).

Although much research effort has been devoted to stabilizing the triplet excited states, the organic phosphorescence lifetime is still less than 10 s. Inspired by inorganic persistent luminescence phosphors based on charge trapping, a charge separation strategy was developed to achieve organic long persistent luminescence (Figure 6). An electron-donating N,N,N′,N′-tetramethylbenzidine (TMB) and an electron-accepting 2,8-bis(diphenylphosphoryl)dibenzo[b,d] thiophene (PPT) were selected to construct the host-guest composite materials (Figure 6A). After an electron is photoexcited from the highest occupied molecular orbital (HOMO)
to the lowest unoccupied molecular orbital (LUMO) of the PPT molecule, an electron is transferred from the TMB LUMO to the PPT LUMO, forming an intermolecular charge-transfer state (Figure 6B). Then the generated PPT anion diffuses through charge hopping among the PPT molecules, which isolates the TMB cation from the counter PPT anion and forms a stable long-lived charge-separated state. Gradual recombination of the PPT anion and TMB cation generates persistent exciplex emission. The green light emission could be detected for more than 30 min from the melt-cast TMB/1,3,5-triphenyl-2-pyrazoline, (TPP) film (Figure 6C).

3.4 Control of vibronic transition path for tunable lasing

One of the most attractive luminescence features of organic materials is their broad emission bands, which are, in theory, very suitable for achieving tunable lasers. However, ruled by the Franck–Condon (FC) principle, organic materials have a high radiative rate and therefore large optical gain at short wavelength, which limits the laser output within the high-energy range, usually corresponding to the 0–1 vibronic transition. To overcome the limitation of the FC principle for tailoring the output of organic vibronic micro/nanolasers over a wide range, a strategy was developed based on the controlled vibronic transition of organic materials (Figure 7A,B). 2,5-Bis(4-biphenylyl)thiophene (BP1T) with strong vibronic coupling was selected as a gain medium owing to its high solid-state luminescence efficiency. 5,5′-bis(4-biphenylyl)-2,2′:5′,2′″-terthiophene (BP3T) with a narrower energy gap was chosen as an optical absorber to modulate the BP1T vibronic emissions (Figure 7B). As shown in Figure 7C, the vibronic emission of BP1T microcrystals can be modulated effectively by changing the doping concentration of light-absorbing materials. As the short-wavelength vibronic emission was suppressed, the luminescence color of BP1T microcrystals gradually changed from bluish green to pure green with the increase of BP3T doping concentration. Meanwhile, the stimulated emission transition in the BP1T microcrystal was switched from the 0–1 to 0–4 vibronic bands (Figure 7D). The output wavelength of organic microlasers was for the first time tailored across all vibronic (0–1, 0–2, 0–3, and even 0–4) bands spanning the whole emission spectrum.

The intrinsic self-absorption effect offers an alternative way to control the vibronic transitions. Because the self-absorption is larger at short emission wavelength and more significant in long propagation distance, the 0–1 lasing was observed in short organic nanowires, while the 0–2 lasing in long ones. Such a self-absorption strategy benefits the gain region broadening of a particular organic gain material, especially toward near-infrared. Besides, a strategy was proposed to switch vibronic transitions in real-time by manipulating the population on the vibronic levels of the ground state. Temperature-controlled dual-wavelength lasing was realized successfully in the organic microcrystals.

3.5 Modulation of energy levels for tunable luminescence/lasing

Another important luminescence feature of organic molecules is their wide spectral coverage. Organic molecular diversity allows for tuning luminescence from ultraviolet to near-infrared via molecular design, which, however,
suffers from difficult and time-consuming synthetic processes. Hence, the ability to tune the luminescence color of one organic molecule continues to grow in importance. The energy levels of organic chromophores are very sensitive to the intermolecular interactions, which can be utilized for tailoring the luminescence color. For example, a cocrystal strategy was developed to tune the luminescence color of a certain organic molecule.\cite{96} As shown in Figure 8A, 1,4-bis-p-cyanostyrylbenzene (A) is a typical fluorescent molecule. The molecule A crystal exhibited a broad yellow excimer emission (Figure 8B, C), which can be attributed to strong $\pi-\pi$ interactions among the chromophores. When a halogenated benzene derivative was introduced to synthesize a cocrystal together with the molecule A, the inter-chromophore distance increases. With the strength of intermolecular interaction decreasing, the excited-state level of the molecule A shifts toward high energy, leading to blue-shifted light emission. With different halogen species, number, and arrangement on the benzene skeleton, the halogenated benzene derivatives B-G exhibited different interactions with the fluorescent molecule A. As different halogenated benzene derivatives induce varied inter-chromophore distances, the luminescence color of the molecule A was tuned from yellow to blue.

To further simplify the luminescence control, a doping method was proposed that needs only a pair of electron donor-acceptor molecules.\cite{97} Incorporation of the electron-donating fullerene (C$_{60}$) into the electron-accepting 1,4-bis(R-cyano-4-diphenylaminostyryl)-2,5-diphenylbenzene (OPV) microcrystals leads to the formation of C$_{60}$-OPV charge-transfer complexes with lower energy levels (Figure 8D). The charge-transfer levels decrease with the increase of the C$_{60}$ doping concentration. As a result, the luminescence color of the C$_{60}$-doped OPV microcrystal gradually changed from green to red when the C$_{60}$ doping concentration increased from 0 to 5.6 mol\% (Figure 8E). Accordingly, the lasing wavelength of the C$_{60}$-doped OPV microcrystal gradually changed from 556 to 607 nm (Figure 8F).

### 3.6 Control of population distribution for tunable luminescence/lasing

Luminescent materials with dynamic wavelength variability are particularly important for applications in chemical and biological sensing, high-density photonic integration, and anti-counterfeiting. Organic molecules with multiple radiative excited states are promising for dynamically tuning luminescence wavelength by controlling the excited-state population distribution.\cite{34,35,36} For example, ICT compounds have two radiative excited states, that is, initially formed LE state and low-lying ICT state (Figure 9A), which is very suitable for achieving dynamically tunable light emission.\cite{58} To make full use of the two upper states, a suitable potential barrier is required to support tailorable population distribution between the LE and ICT states. As shown in Figure 9B, trans-4-[4-(dimethylamino)styryl]-1-methylpyridinium iodide (DASPI) is a typical ICT organic compound. \(\beta\)-Cyclodextrin (\(\beta\)-CD) with cavity dimension similar to the DASPI molecular size to synthesize the DASPI@\(\beta\)-CD
inclusion complex. The β-CD provides a partial restriction on the rotation of the DASPI excited state, producing a moderate potential barrier between the LE and ICT states. Therefore, the excited-state population can be readily transferred from the LE to the ICT state by elevating temperature. With the temperature increasing from −60 to 120°C, the DASPI@β-CD microcrystal exhibit a gradual color change from yellow to red, corresponding to an emission wavelength redshift from 570 to 622 nm (Figure 9C). Accordingly, the lasing wavelength of a DASPI@β-CD microcrystal is tuned dynamically and continuously from 570 to 620 nm (Figure 9D).

3.7 Energy transfer for tunable luminescence/lasing

Intermolecular energy transfer in organic composite materials composed of multiple chromophores has enabled many important applications in chemical sensing, tunable luminescence, optical computing, and multicolor lasing. So far, diverse organic composite materials have been synthesized, exhibiting energy transfer and consequent composition-dependent light emission.[43,98,99] It is noteworthy that the white organic light-emitting materials and devices are particularly appealing for their wide applications in full-color display and low-energy-consumption lighting. Proper control of the energy transfer from blue-cyan to orange-red light-emitting organic dyes can lead to white light emission.[100–103]

In recent years, mesoscopic organic composite crystals have attracted much research attention because of their great potential in on-chip photonic integration.[32,38] As shown in Figure 10A, a blue (TPP) and an orange (rubrene) light-emitting organic molecule were selected as energy donor and acceptor, respectively, to construct luminescence tunable composite microcrystals.[104] The absorption spectrum of rubrene shows good overlap with the fluorescence spectrum of TPP in the 400–550 nm wavelength range (Figure 10B), which would result in efficient energy transfer in the doping systems. The binary nanomaterials of the TPP and rubrene with controllable contents were fabricated through adsorbent-assisted physical vapor deposition (Figure 10C). With the rubrene content increasing from 0% to 100%, the luminescence color of the nanostructures gradually changed from blue to orange (Figure 10C,D), which is attributed to the efficient FRET process. White light-emitting can be achieved when the TPP/rubrene molar ratio is proper (100:1). Furthermore, these one-dimensional binary nanomaterials can serve as active optical waveguides, which inspire many important works on the use of one-dimensional organic composite structures in waveguide sensors,[56] modulators,[49,105–108] and transistors.[67,109]

Organic FRET material systems have exhibited many advantages in constructing solid-state lasers. For example, the organic FRET composite materials are four-level laser systems, which are more efficient than the quasi four-level laser systems of organic single-component materials. As a result, the FRET-assisted organic lasers have low threshold pump density and high operation stability.[110,111] Another advantage of the FRET materials systems lies in the laser output flexibility. The laser output wavelength can be extended across the whole visible light region by simply changing/adding energy-accepting laser molecules.[40,112–116] Besides, the FRET-assisted organic lasers supported dual-wavelength output, and the output can be switched dynamically by modulating the FRET process.[117,118]

4 CONCLUSION AND OUTLOOK

Organic composite materials have enormously advanced the field of luminescent materials. Over the past two decades, a wide variety of luminescent organic composite materials have been synthesized, which resulted in lots of intriguing
light-emitting phenomena by manipulating the molecular excited states. As the field is still rapidly growing, many new opportunities and challenges are ahead of us. First, the library of luminescent organic composite materials and architectures will continue to expand. The diversity of organic molecules and intermolecular interactions enables us to keep exploring novel organic composite materials with desired compositions and structures toward practical applications. Second, more mechanisms for manipulating the excitonic excited states based on the component combination should be explored to generate new light-emitting properties. Third, the application scope of the component combination strategy should be extended to manipulate more kinds of excited-state structures and dynamics, such as doublet states and spin polarization. In such composite materials, new photophysical knowledge and photonic applications are expected.

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ETHICS STATEMENT

This review article does not involve any human investigation and animal experiment.

DATA AVAILABILITY STATEMENT

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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