

A Fluorescence–Phosphorescence–Phosphorescence Triple-Channel Emission Strategy for Full-Color Luminescence

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Organic luminogens constitute promising prototypes for various optoelectronic applications. Since gaining distinct color emissions normally requires the alternation of the conjugated backbone, big issues remain in material synthetic cost and skeleton compatibility while pursuing full-color luminescence. Upon a facile one-step coupling, three simple but smart perchalcogenated (O, S, and Se) arenes are synthesized. They exhibit strong luminescent tricolor primaries (i.e., blue, green, and red, respectively) in the solid state with a superior quantum yield up to >40% (5-10 times higher than that in corresponding solutions). The properties originate from a fluorescencephosphorescence-phosphorescence triple-channel emission effect, which is regulated by S and Se heavy atoms-dependent intersystem crossing upon molecular packing, as well as Se-Se atom interaction-caused energy splittings. Consequently, full-color luminescence, including a typical white-light luminescence with a Commission Internationale de l'Eclairage coordinate of (0.30, 0.35), is realized by complementarily incorporating these tricolor luminescent materials in the film. Moreover, mechanochromic luminescent color conversions are also observed to achieve the fine-tuning of the luminescent tints. This strategy can be smart to address full-color luminescence on the same molecular skeleton, showing better material compatibility as an alternative to the traditional multiple-luminophore engineering.

the development of solid-state organic optoelectronics.^[2] It is by now well-known that it is possible to design and synthesize luminophores with different conjugated backbones to regulate emissions covering distinct visible-light spectral regions, for finally achieving the three primaries or even full-color luminescence in solution, in the solid state and in devices.^[3] Fortunately, this principle can also be harnessed into different types of molecular design, to reveal diverse luminescence colors in pertinent light-emitting applications.^[4] Nevertheless, these regulation strategies for emission color usually lack the skeleton compatibility from the perspective of the conjugated backbone alternation. This issue may reduce the service life of a system or bring in other unexpected practical problems. Therefore, it remains challenging but desirable to develop a simpler strategy to achieve full-color luminescence regulation, and meantime to minimize the material synthetic cost and incompatibility.

Fluorescence-phosphorescence regulation has been a promising luminescence controlling strategy in recent years, and

Organic luminogens, especially those that can emit brightly in the solid state, exhibit great application potential in organic light-emitting diode, lighting, information storage, and imaging technologies.^[1] In 2001, Tang et al. discovered the aggregationinduced emission phenomenon, which further strengthened has generalized a concept to fully utilize different emissive pathways of spin multiplicity.^[5] Although radiative pathways from triplet states are generally forbidden, scientists have still presented plenty of examples of room-temperature phosphorescence emitters, in which the intersystem crossing (ISC) process

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is boosted by the internal or external heavy atom effect.^[6] For a single molecular skeleton, room-temperature phosphorescence, and fluorescence can cover individual visible-light spectral regions because of the significant singlet–triplet energy difference.^[7] Therefore, employing such an energy difference, and then to further control the fluorescence–phosphorescence proportion and wavelength by structural means or molecular aggregation, might be an effective way to address the abovementioned hypothesis.

Recently, persulfurated aromatic compounds (i.e., persulfurated arenes) have been found to show attractive and peculiar photophysical properties regarding room-temperature phosphorescence.^[8] Multiple sulfur atoms in a molecular skeleton facilitate ISC and spin-orbit coupling (SOC) effects, while the asterisk-type structures can allow the suppression of molecular movements and the nonradiative decay upon aggregation.^[9] This kind of compounds can be easily synthesized by low-cost and commercial starting materials.^[10] Our group has developed a series of methods to achieve fluorescence-phosphorescence regulation of persulfurated arenes upon diverse self-assemblies.^[11] In this work, we further broaden the use of such asterisk structures by chalcogen chemistry, for which three arene molecules containing O, S, and Se atoms have been designed and synthesized by one-step coupling reaction economically (see the chemical structures in Figure 1). The synthetic routes and details for these compounds are outlined in the Supporting Information. In this design, O atom is taken for the consideration of a fluorescence emission due to the lack of heavy atom effect. Both S and Se atoms can strengthen ISC for phosphorescence, but their different chalcogen interactions might regulate the phosphorescence wavelength. Thus, a unique fluorescence-phosphorescence-phosphorescence triple-channel emission strategy can be established to fulfil the full-color luminescence hypothesis (see below for details). Upon obtaining the perchalcogenated arene samples, we eventually find that they distinctly exhibit the three luminescent primaries (blue, green, red), respectively.



Figure 1. Illustration of the chemical structures and the one-step coupling of the perchalcogenated arenes, where X stands for the element O, S, and Se in compounds 1, 2, and 3, respectively. These compounds correspondingly exhibit strong luminescent primaries (blue, green, red) in the solid state.

Three of the compounds all show local excited states in solution at room temperature,^[12] as monitored by the fact that their absorption and fluorescence wavelengths are very closely similar to each other in different solvents (Figure S1, Supporting Information). At room temperature conditions, fluorescence can be uniquely observed in the solutions, since photoluminescent lifetimes of these bands from 400 to 500 nm are always on the nanosecond scale (Figures S2–S4, Supporting Information). These solutions show relatively low quantum yields (QY, \approx 3.1%, 5.2%, and 3.0% for compounds 1, 2, and 3, respectively). Upon lowering the temperature to 77 K, triplet emission pathways of compound 2 and 3 can be opened up while long-lifetime phosphorescence bands redshifted to over 500 nm (Figures S6 and S7, Supporting Information). The SOC effects in the Secontaining compound 3 are so strong that even simultaneous phosphorescence from T_1 and T_2 states can be observed in dichloromethane solution at 77 K, which is in excellent agreement with quantum-chemical calculations (Figure S8, Supporting Information). There remains no phosphorescence stemming from compound 1 at low temperature (Figure S5, Supporting Information), because of not only the light O atoms that are unable to strengthen the ISC process (SOC effects are too weak), but also the quite large S_1-T_1 gap (1.5 eV) followed by our quantum-chemical calculations (Figure S8, Supporting Information).

These perchalcogenated arenes exhibit bright solid-state luminescence in the film states (Figure 2). Interestingly, they emit the three distinct luminescent primaries (blue, green, red), respectively, like their corresponding solid sample. Compound 1 shows a blue emission with two main fluorescence peaks at around 426 and 470 nm, featuring distinct singlet excited vibrational states. For compound 2, the fluorescence emission is completely suppressed and a strong phosphorescence band at 513 nm with long lifetime is found, corresponding to green emission. For compound 3, in addition to the band over 500 nm, a relatively strong phosphorescence band at ≈600 nm also appears, further adjusting the tint to make it produce red emission. All the three films display similar emission properties at 77 K, except showing prolonged lifetime of those phosphorescence bands (Figure S9, Supporting Information). This indicates that competitively nonradiative factors are largely inhibited at low temperature. Therefore, chalcogen chemistrybased organic emitters that straightforwardly give rise to three luminescent primaries could be achieved. The QY of these three compounds in the solid state were measured as 8%, 42%, and 21%, respectively, which were 5-10 times higher than that in corresponding solutions and featured these arenes were superior solid-state luminescent materials.

To better clarify such a unique behavior in the solid state, we have crystallized compounds 2 and 3 to explore their peculiar phosphorescence properties with the aid of a single-crystal conformational analysis. Analogously to their emissions in the film, the emission spectra of crystal 2 and 3 show, respectively, a single phosphorescence band (\approx 523 nm) and double phosphorescence bands (\approx 542 and \approx 622 nm, although the proportion differs, see **Figure 3**a,d). Meanwhile, the crystal structures of the two compounds differentiated greatly. The crystal of compound 2 comprised a single conformation in a unit cell, whereas crystal 3 involved two conformations in





Figure 2. Photophysical properties at room temperature. Emission spectra of the films of a) Compound 1 (λ_{ex} = 330 nm), b) Compound 2 (λ_{ex} = 425 nm), and c) Compound 3 (λ_{ex} = 470 nm) prepared from *N*,*N*-dimethylformamide (DMF), respectively. Photoluminescent lifetime of compound 1 measured at d) 426 nm and e) 470 nm emission, of compound 2 measured at f) 513 nm emission, and of compound 3 measured at g) 540 nm and h) 600 nm emission, respectively.

the same unit cell (Figure 3b,e). These conformations are symmetric as monitored by the typical bond angles (Figure 3), and we can assume that these distinct conformational interactions largely determine their photoluminescent properties. It is easily acceptable that the band at around 520–540 nm is an ordinary T_1 phosphorescence close to the emission band in solution study.^[8,9] On the other hand, the mutual control study in this

work indicates that the second band at 620 nm in crystal 3 most likely corresponds to phosphorescence from a Se-dependent molecular interaction.^[13]

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To gain further insight into the dual phosphorescence feature of compound 3 in the solid state, density functional theory (DFT) calculations were employed to study the energy relationships upon the geometries extracted from the single crystal



Figure 3. Single-crystal luminescence and conformational peculiarities. Emission spectra upon 365 nm excitation in crystals of a) Compound 2 and d) Compound 3, respectively. Unit cell display in crystals of b) Compound 2 and e) Compound 3, respectively. Packing display in crystals of c) Compound 2 and f) Compound 3, respectively. Typical C–S–C and C–Se–C bond angles are highlighted.



Figure 4. Chalcogen interaction analysis in the crystals and theoretical study for a) Compound 2 and b) Compound 3. The splitting of frontier molecular orbitals in a dimeric model of the two molecules was calculated by the DFT B3LYP/6-311G(d) method in a single point. The geometries were extracted from the X-ray analysis data. The shape of HOMO and LUMO orbitals of the single molecules is independent to the starting geometry (extracted from X-ray, optimized in S₀ or T₁ electronic state). The same trend was also verified by TD-DFT calculations.

X-ray diffraction (XRD) data (Figures S11-S14, Supporting Information). The two conformations coexisting in a single unit cell of crystal 3 signify that Se atoms can interact with each other between the neighboring molecules to behave a dimeric conformation (see the Se-Se contacts of 3.85 and 4.58 Å, in consistency with the van der Waals (VdW) radius of Se (1.9–2.2 Å), Figure 4b). In contrast, the closest S-S contact upon molecular packing is about 6 Å (Figure 4a) that is much bigger comparing with the double VdW radius of S ($1.8 \times 2 = 3.6$ Å). A frontier molecular orbitals study shows that the highest occupied molecular orbital (HOMO) orbital for both compound 2 and 3 is purely n-orbital localized on the S or Se atoms. The overlap between these orbitals upon Se-Se interaction produces the splitting of HOMO orbital upon a corresponding dimeric conformation, relative to the monomeric state in compound 3 (Figure 4). This behavior does not occur in compound 2. The HOMO-1 is bonding with respect to the Se-Se interaction, while the HOMO is antibonding which provides its destabilization (4.77 vs 4.86 eV in the monomeric state). The lowest unoccupied molecular orbital (LUMO) orbital is a typical π -orbital localized on the inner phenyl core. These orbitals cannot interact with each other in the crystal phase of 2 or 3, and indicate no energy splitting. In this way, with the splitting of the HOMO in crystal 3, we can observe $n-\pi$ emission at 600 nm connected to the dimeric conformation upon the Se-Se interaction, and at 540 nm owing to the original intrinsic emission. The proportion of the two emission bands is adjustable due to the pattern and degree of dimeric packing. In this way, the disappearance of the band at around 600 nm in solution is accordingly expected, since there is no such a packing behavior (Figure S7, Supporting Information). This effect could also be proved on doping of compound 3 into poly(methylmethacrylate) (PMMA) film. The isolation of compound 3 molecules by PMMA matrix can result in the weakening of the Se-Se interaction, leaving the intrinsic phosphorescence band at ~540 nm maintained but the one from the dimeric conformation at ≈600 nm diminished (Figure S10, Supporting Information). Regarding compound 2, we did not observe such a dual-band phosphorescence due to the inefficient S-S interactions in the crystal.

As the luminescent color of compounds 1–3 related to blue, green, and red primaries, respectively, we addressed the fullcolor luminescence by blending these emitters with different controlled ratios. The Commission Internationale de l'Eclairage (CIE) color coordinates of the emission spectra of compounds 1-3 were calculated to be (0.13, 0.15), (0.26, 0.63), and (0.52, 0.48), from the corresponding emission spectra (Figure 2), which reside at the blue, green, and red emission region, respectively. After complementarily blending two of these compounds to prepare the film samples of mixture (1, 2), (2, 3), and (1, 3), we can obtain films with the luminescent color of cyan (0.19, 0.46), yellow (0.41, 0.55), and purple (0.28, 0.22), respectively. When mixing all the three compounds complementarily, we can get a film with the luminescent color located in the white-light region (0.27, 0.34). These CIE coordinates were also calculated from their corresponding film emission spectra of the mixtures (Figure S15, Supporting Information). The XRD spectra of these full-color films (Figure S16, Supporting Information) show that the cyan, purple, and white films from mixture (1, 2), (1, 3), and (1, 2, 3) were crystalline, while the yellow film from mixture (2, 3) was amorphous. The XRD spectra of compounds 1, 2, 3 were also shown in Figures S17-S19 (Supporting Information), respectively.

Since lots of molecular packing-dependent luminescence is very sensitive to mechanical stimuli,^[14] a mechanochromic luminescent color change can also be observed in our materials. After grinding, we can find that CIE luminescence coordinates of the film samples of the mixtures change to a certain degree (see the 4'-7' dots in Figure 5). It can be found that the emission bands from the solid film are a little broader than those of the single crystal (see Figures 2 and 3), indicating that disordering in our materials can broaden the emission peak. Our sample can become more amorphous upon mechanical stimuli. Therefore, all these samples basically exhibit a whitening effect upon grinding, resulting from the broadening of the luminescence bands upon the amorphous tendency (see the emission spectra with grinding in Figure S20, Supporting Information). Especially, the CIE coordinate of the film state of mixture (1, 2, 3) turned to be (0.30, 0.35) after grinding, which www.advancedsciencenews.com

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Figure 5. CIE 1931 chromaticity diagram. The dots 1–3 signify the luminescent color coordinates for the corresponding film states of compound 1–3, respectively. The dots 4–6 feature the luminescent color coordinates for the film states of mixture (1, 2), (2, 3), and (1, 3), respectively. The dot 7 signifies the luminescent color coordinates for the film state of mixture (1, 2, 3). The dots 4'–7' feature the luminescent color coordinates for the corresponding ground film states.

is very close to the coordinate (0.33, 0.33) of the standard whitelight illumination. These results suggest that the fine tuning of the luminescent tints of our materials can be realized by external stimuli, and that such a mechanochromic luminescent behavior can be recovered by fuming. This further indicates the facilitation and controllability of the presented strategy.

Grinding

Fuming

(0.19, 0.46)

(0.23, 0.40)

In summary, a fluorescence-phosphorescence-phosphorescence strategy upon molecular packing was demonstrated to prepare organic emitters for full-color luminescence. Perchalcogenated (O, S, and Se) arenes, prepared by a simple but smart chalcogen-based chemistry, can generate bright luminescent primaries (i.e., blue, green, and red) in the solid state on the same skeleton, whereby a chalcogen (mainly the S and Se) heavy atom controlled intersystem crossing upon molecular packing, as well as a Se-Se atom interaction caused energy splittings, played key roles. Full-color luminescence in the film was achieved by complementarily mixing these tricolor luminescent materials. Meanwhile the fine tuning of the luminescent tints can be manipulated by mechanical stimuli. We believe that the strategy demonstrated herein can be valuable for future constructing low-cost and material compatible optoelectronic devices and equipment.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

 ${\sf crystal}$ engineering, luminescence, molecular packing, noncovalent interactions, phosphorescence

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