

基于卤素取代邻苯二甲酰亚胺新型有机发光材料的合成及其性能研究

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摘要 方便地合成了三个含有卤素取代邻苯二甲酰亚胺与咔唑基团的新型有机发光材料 **Br-Al-Cz**, **Cl-Al-Cz** 和 **F-Al-Cz**, 发现它们不仅具有强的聚集诱导发光效应, 而且显示膜态下热激活延迟荧光以及晶态诱导的室温磷光性质. 尤其是化合物 **Br-Al-Cz** 表现出肉眼可见的长余辉室温磷光现象, 因此在数据加密等中具有潜在用途.

关键词 有机发光材料; 室温磷光; 热激活延迟荧光; 聚集诱导发光; 邻苯二甲酰亚胺

Synthesis and Properties of New Organic Luminescent Materials Based on Halogen-Substituted Phthalimides

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Abstract A new kind of organic luminescent materials containing halogen-substituted phthalimide group and carbazole subunit have been conveniently synthesized. They not only showed strong aggregation-induced emission (AIE) effect, but also exhibited thermally activated delayed fluorescence (TADF) in films and crystallization-induced room-temperature phosphorescence (RTP) properties. Especially, RTP with marked afterglow for **Br-Al-Cz** was observed by naked eye, which could be used as the promising smart materials for the encryption application.

Keywords organic luminescent material; room-temperature phosphorescence; thermally activated delayed fluorescence; aggregation-induced emission; phthalimide

1 Introduction

Pure organic materials with room-temperature phosphorescence (RTP) properties have attracted much attention for their potential applications in anti-counterfeiting, molecular sensing and display.^[1] In particular, phosphorescence can eliminate background fluorescence interference through the time-gate technique in the biological field.^[2] However, most of phosphorescence materials are confined to the inorganic or precious organometallic counterparts, which have distinct shortcomings, such as biological toxicity, high cost and so forth.^[3,4] Developing efficient pure organic RTP materials still remain a great challenge owing to the weak spin-orbit coupling (SOC) and the subsequent

low intersystem crossing (ISC) rate to yield triplet from S₁ state. The introduction of heavy halogens can generally increase the SOC for efficient ISC.^[5] Crystallization-induced phosphorescence (CIP) in pure organic luminogens is also an effective method to obtain organic RTP materials.^[6] Orderly molecular packing can rigidify the molecular conformation through effective intermolecular interactions in the crystal states, including multiple intermolecular hydrogen bonding,^[7] halogen bonding^[8] and Vander Waals force.^[9] Thus, the key issue for the development of efficient and pure organic RTP materials is how to promote intersystem crossing (ISC)^[10] and suppress non-radiative decay.^[11]

Thermally activated delayed fluorescence (TADF) is a

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special radiative transition based on converting from the lowest triplet excited state (T_1) to the lowest singlet excited state (S_1) by effective reverse intersystem crossing (RISC) process.^[12] TADF materials can theoretically realize 100% internal quantum efficiency for high-efficiency and stable OLED emitters.^[13] Therefore, TADF materials are the promising third-generation organic luminescent materials after fluorescent and phosphorescent materials. In recent years, the organic luminogens with dual emissions of RTP and TADF have attracted increasingly interest for their potential optoelectronic applications as well.^[14] However, the reported purely organic luminogens with dual emissions of RTP and TADF were preferred twisted donor-acceptor conjugation systems, and they generally showed short lifetime of phosphorescence. Moreover, the examples on the luminogens with dual properties of RTP with afterglow and TADF have been rarely known. Herein, a new kind of organic luminescent materials **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** containing halogen-substituted phthalimide group and carbazole subunit was reported (Figure 1), which not only showed strong aggregation-induced emission (AIE) effect,^[15] but also exhibited crystallization-induced RTP and TADF properties in films. Especially, RTP with persistent afterglow for **Br-AI-Cz** could be observed by naked eye, which would be used as the promising smart materials for the encryption application.

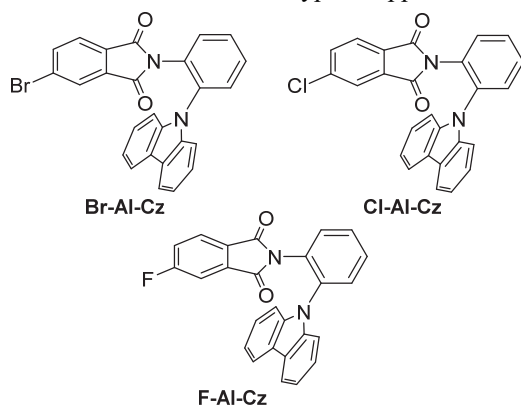
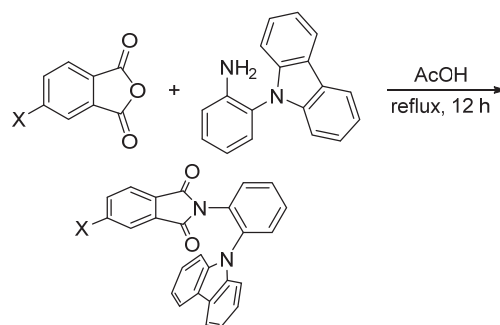


Figure 1 Structures of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz**

2 Results and discussion

By the condensation of 5-halogen-phthalic anhydride and 2-(9*H*-carbazol-9-yl)aniline in glacial acetic acid, followed by cooling to room temperature and then filtering the solids from the solution, **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** could be easily synthesized in 85%, 80% and 82%, respectively, without further purification (Scheme 1). Their structures were confirmed by ^1H NMR, ^{13}C NMR, HRMS spectra and single-crystal X-ray structure.

Br-AI-Cz, **Cl-AI-Cz** and **F-AI-Cz** showed a broad absorption band ranging from 331~335 nm, which were assigned to π - π^* transitions (Figure 2a). As shown in Figure 2, **Br-AI-Cz** showed very weak emission in pure tetrahydrofuran (THF). However, when the value of water fraction reached to 90%, a relatively strong fluorescence emission of the solution was observed owing to aggrega-



Br-AI-Cz: X = Br 85%; **Cl-AI-Cz**: X = Cl, 80%; **F-AI-Cz**: X = F, 82%

Scheme 1 Synthesis of the target compounds

tion of the molecules. With the continuous increase of water fraction, the fluorescence intensity enhances gradually. When the value of water fraction reaches to 95%, the fluorescence intensity of **Br-AI-Cz** was 32.4-fold relative to that in pure THF when the value of water fraction reaches to 95%, which showed the strong AIE effect. Similarly, **Cl-AI-Cz** and **F-AI-Cz** also showed the AIE properties. Compared with those ones in THF, the fluorescence intensity of **Cl-AI-Cz** increased 28.5-fold, while the fluorescence intensity of **F-AI-Cz** increased 26-fold when the value of water fraction reaches to 95%.

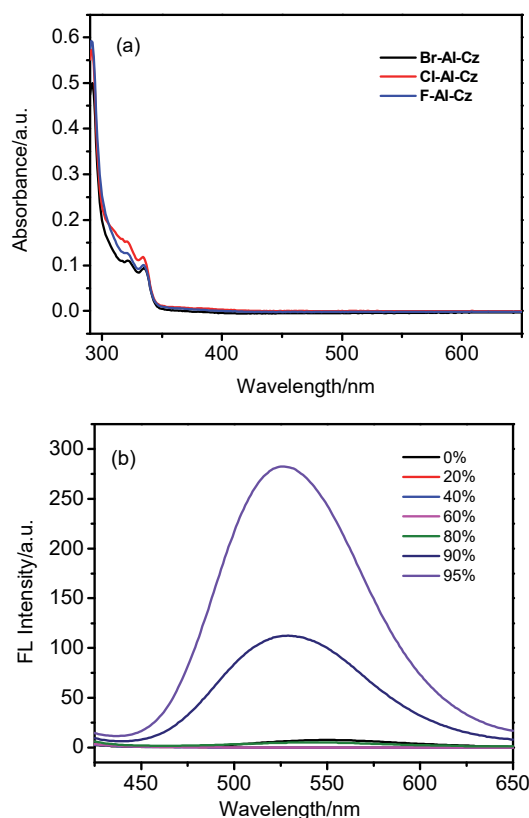


Figure 2 (a) Absorption spectra of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** in toluene ($c=1.0 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$), (b) FL spectra of **Br-AI-Cz** in THF and THF/water mixtures with different f_w values ($c=1.0 \times 10^{-5} \text{ mol}\cdot\text{L}^{-1}$)

The optimized geometry and the electron density distribution of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** were investigated by density functional theory calculations with

B3LYP/6-31G* basis set. Three compounds all showed the separated HOMO and LUMO distributions on their optimized geometries. The HOMOs are predominantly located on the electron-donating carbazole group, whereas the LUMOs are distributed over the electron-withdrawing halogen-phthalimide group. The frontier molecular orbital of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** showed small overlap mainly on the donor and acceptor units, resulting in the appreciable ΔE_{ST} values, evidenced by the calculated ΔE_{ST} values of 0.068, 0.032 and 0.031 eV, respectively. Small ΔE_{ST} values led to high efficiency of the RISC, which could induce the TADF capability. In order to demonstrate the TADF properties of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz**, their transient photoluminescence (PL) decay spectra were further measured in film states, respectively (Figure 3). It was found that **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** displayed a distinctive microsecond-scaled delayed relaxation at room temperature, which was assigned to TADF. To further confirm the TADF properties of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz**, the time-gated measurement was carried out. The normalized spectra with a delay time of 5 s overlapped well with the steady-state spectra, which suggested that the long-lived emissions with the lifetimes in the microsecond range were the TADF emissions.

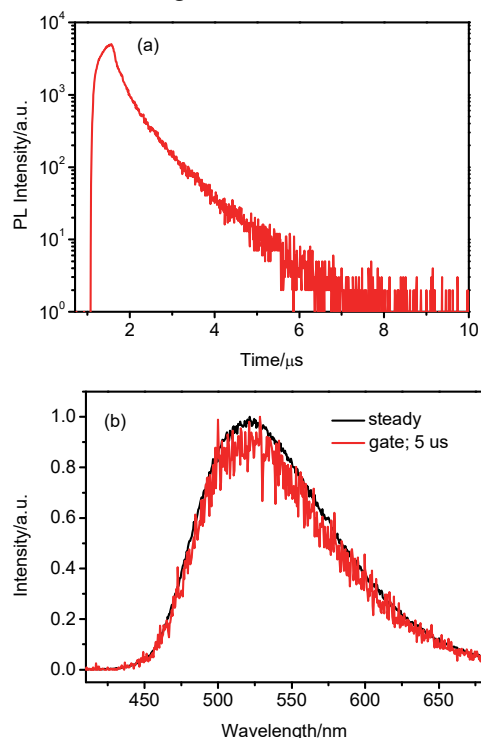


Figure 3 (a) Transient PL characteristics of **Br-AI-Cz** at room temperature in film state, (b) normalized steady-state and time-gated emission spectra of **Br-AI-Cz**, in film state

It was further found that the three molecules exhibited obvious RTP characteristics in the crystalline states. For **Br-AI-Cz**, its crystals emitted sky blue fluorescence and yellow phosphorescence with the detailed structure at room temperature before and after removal of the excitation source (Figure 4a), which might be due to its relatively

large ΔE_{ST} . The fluorescence commission internationale de L'Eclairage (CIE) coordinate (0.16, 0.33) and phosphorescence CIE coordinate (0.31, 0.54) at room temperature were found (Figure 4b). For **Cl-AI-Cz** and **F-AI-Cz**, their fluorescence CIE coordinates were found to be at (0.16, 0.32) and (0.15, 0.26), respectively. Simultaneously, their phosphorescence CIE coordinates (0.22, 0.48) and (0.19, 0.50) were obtained at room temperature, respectively. To further obtain insight into emission of the crystals, the transient PL decay curve of **Br-AI-Cz** was measured in the crystalline state, the phosphorescence lifetime was calculated to be 28.7 ms. The transient PL decay curves of **Cl-AI-Cz** and **F-AI-Cz** were also measured in the crystal states, the phosphorescence lifetimes were found to be 12.3 ms and 9.1 s, respectively. The results indicated that the intramolecular motions were efficiently restricted in the crystalline states to prevent the loss of excited-state energy through non-radiative decay pathway.

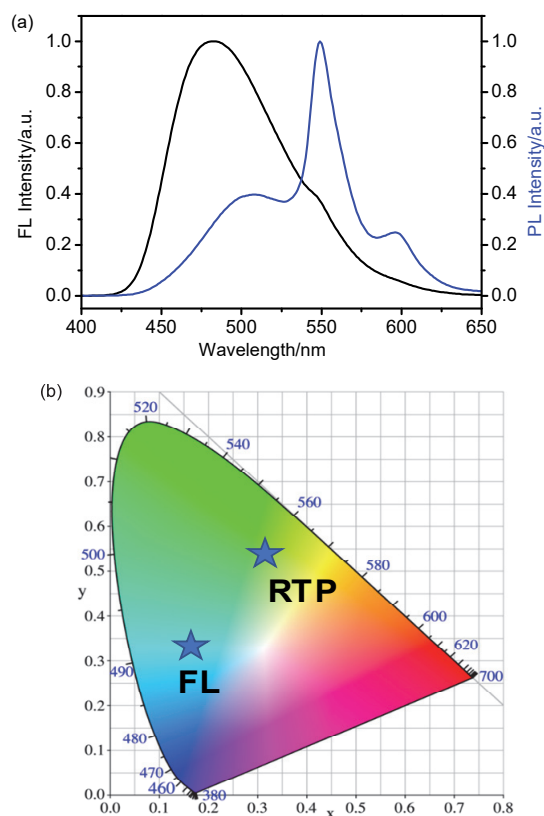


Figure 4 (a) Normalized fluorescence (black, λ_{ex} = 340 nm) and phosphorescence (blue, delay time of 0.005 ms and λ_{ex} = 340 nm) spectra of **Br-AI-Cz** at room temperature in the crystalline state, and (b) CIE coordinates of **Br-AI-Cz** in the crystalline states

To obtain more insights into persistent RTP about molecular conformations and crystal packing of **Br-AI-Cz**, its single crystal structure was obtained (Figure 5). It was found that the carbazole group in **Br-AI-Cz** unit adopted a roughly parallel stacking to the carbazole group of neighbouring molecules with a short distance of 0.3324 nm, which is beneficial to suppress nonradiative transition of triplet excitons.^[16] The crystal structure also showed that the dihedral angle of carbazole group and imide moiety

was about 56.71° . This small dihedral angle made the carbazole group and imide moiety much closer together, which might result in the stronger $\pi\cdots\pi$ (0.3443 and 0.3258 nm) interactions. Through C—H $\cdots$ O (0.2345 and 0.2560 nm) and C—H $\cdots\pi$ (0.2803 nm) intermolecular interactions, the molecules could further form a 3D network structure, which might be powerful to restrict molecular motions and suppress the non-radiative decay. Besides the heavy halogens to increase the SOC for efficient ISC, the efficient and persistent RTP for **Br-AI-Cz** could be achieved by the utilization of crystallization and a rigid matrix to suppress the nonradiative relaxation of the excited state as well.

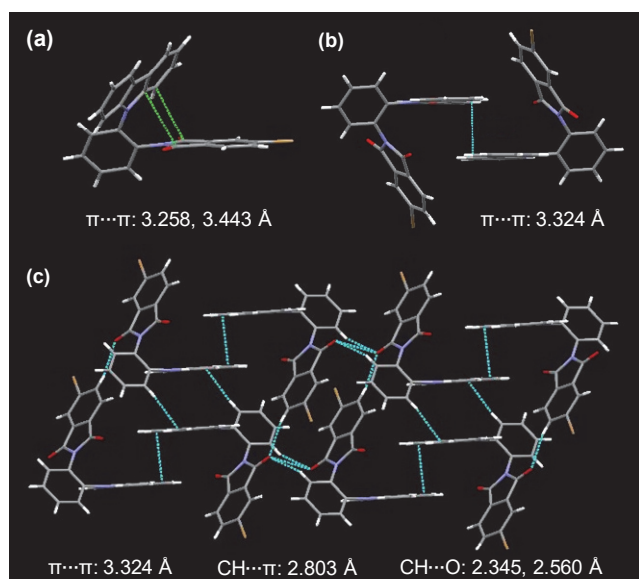


Figure 5 (a) Single crystal structure of **Br-AI-Cz**, (b) the $\pi\cdots\pi$ intermolecular interaction, and (c) the stacking interactions of the adjacent molecules.

The RTP materials with different lifetimes of **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** made them promising materials for data encryption and decryption. As shown in Figure 6, we arranged a character “8” using **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** samples in crystalline states. In the presence of UV light, the character “8” was visible. After removal of the UV light for 0.2 s, the character “7” was visible.

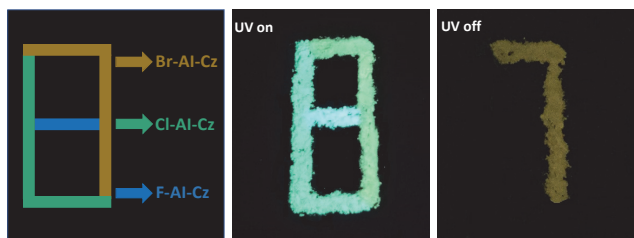


Figure 6 Illustration of the character “8” arranged by **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** in the crystalline states under UV irradiation and after removal of ultraviolet lamp during the afterglow decay process

3 Conclusions

In summary, a new kind of organic luminescent

materials **Br-AI-Cz**, **Cl-AI-Cz** and **F-AI-Cz** containing halogen-substituted phthalimide group and carbazolyl subunit have conveniently been synthesized, and then it was demonstrated that the luminogens all not only showed AIE and TADF properties, but also exhibited the crystallization-induced RTP properties. Especially, it was found that RTP with markedly afterglow for **Br-AI-Cz** could be observed by naked eye, which would be used as the promising smart materials for the advanced-encryption application.

4 Experimental section

The reagents were all purchased from commercial sources and used without further purification. 2-(9*H*-Carbazol-9-yl)aniline was prepared according to the literature procedure.^[13c]

4.2 Synthesis of Br-AI-Cz

A mixture of 5-bromoisobenzofuran-1,3-dione (454 mg, 2 mmol) and 2-(9*H*-carbazol-9-yl)aniline (516 mg, 2 mmol) in glacial acetic acid (5 mL) was refluxed for 12 hours. The mixture was slowly cooled to room temperature. Then, the solids were filtered from the solution, and product (792 mg, 85 %) was obtained as light yellow solid. m.p. $201\sim 202^\circ\text{C}$. ^1H NMR (300 MHz, CDCl_3) δ : 7.99 (d, $J=7.7$ Hz, 2H), 7.73 (s, 1H), 7.71 $\sim$ 7.55 (m, 5H), 7.46 (d, $J=7.9$ Hz, 1H), 7.35 $\sim$ 7.30 (m, 2H), 7.25 $\sim$ 7.15 (m, 4H); ^{13}C NMR (126 MHz, CDCl_3) δ : 165.4, 164.9, 140.6, 137.1, 135.4, 132.9, 130.5, 130.3, 130.0, 129.8, 129.3, 129.0, 128.9, 126.8, 126.0, 126.0, 124.9, 123.4, 123.4, 120.2, 120.2, 120.1, 110.0; HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{15}\text{BrN}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 467.0317, found 467.0384.

4.3 Synthesis of Cl-AI-Cz

A mixture of 5-chloroisobenzofuran-1,3-dione (1.27 g, 7 mmol) and 2-(9*H*-carbazol-9-yl)aniline (1.81 g, 7 mmol) in glacial acetic acid (7 mL) was refluxed for 12 h. The mixture was slowly cooled to room temperature. Then the solids were filtered from the solution, and product (2.36 g, 80 %) was obtained as white solid. m.p. $199\sim 200^\circ\text{C}$. ^1H NMR (300 MHz, CDCl_3) δ : 7.98 (d, $J=7.7$ Hz, 2H), 7.72 $\sim$ 7.54 (m, 5H), 7.51 (d, $J=4.4$ Hz, 2H), 7.35 $\sim$ 7.30 (m, 2H), 7.27 $\sim$ 7.13 (m, 4H); ^{13}C NMR (126 MHz, CDCl_3) δ : 165.3, 165.0, 140.7, 140.6, 135.4, 134.1, 133.0, 130.5, 130.3, 130.0, 129.4, 129.4, 129.0, 126.0, 126.0, 124.7, 123.9, 123.4, 123.4, 120.2, 120.2, 120.1, 110.0. HRMS (ESI) calcd for $\text{C}_{26}\text{H}_{15}\text{ClN}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 423.0822, found 423.0888.

4.3 Synthesis of F-AI-Cz

A mixture of 5-fluoroisobenzofuran-1,3-dione (830 mg, 5 mmol) and 2-(9*H*-carbazol-9-yl)aniline (1.29 g, 5 mmol) in glacial acetic acid (5 mL) was refluxed for 12 h. The mixture was slowly cooled to room temperature. Then, the solids were filtered from the solution, and product (1.66 mg, 82%) was obtained as white solid. m.p. $186\sim 187^\circ\text{C}$; ^1H NMR (300 MHz, CDCl_3) δ : 7.98 (d, $J=7.7$ Hz, 2H), 7.69 $\sim$ 7.58 (m, 5H), 7.38 $\sim$ 7.11 (m, 8H); ^{13}C NMR (126

MHz, CDCl₃) δ : 167.2, 165.2, 164.9, 164.9, 140.6, 135.4, 134.2, 134.1, 130.5, 130.3, 130.0, 129.4, 128.9, 127.1, 127.1, 126.0, 126.0, 125.9, 125.9, 123.4, 121.2, 121.1, 120.2, 120.1, 120.0, 111.3, 111.1, 110.0. HRMS (ESI) calcd for C₂₆H₁₅BrN₂O₂ [M + H]⁺ 407.1118, found 407.1183.

4.4 X-ray crystallography

CCDC 1917991 (**Br-AI-Cz**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre.

Crystal data for **Br-AI-Cz**: C₂₆H₁₅BrN₂O₂, M_w = 467.31, crystal size 0.25 mm $\times$ 0.25 mm $\times$ 0.212 mm, triclinic, space group *P*1, a = 0.83236(3) nm, b = 0.83566(2) nm, c = 1.48856(4) nm, α = 87.095(2)°, β = 86.130(2)°, γ = 75.506(2)°, V = 0.99955(5) nm³, Z = 2, ρ_{calcd} = 1.553 Mg/m³, T = 170.01(10) K, μ = 3.022 mm⁻¹, 11932 reflections measured, 3932 unique [$R(\text{int})$ = 0.0157], final R indices [$I > 2\sigma(I)$]: R_1 = 0.0353 and wR_2 = 0.0896, R indices (all data): R_1 = 0.0385 and wR_2 = 0.0944, absorption correction: semi-empirical from equivalents.

Supporting Information ¹H NMR and ¹³C NMR spectra, photophysical properties, theoretical calculations, cartesian coordinates. The Supporting Information is available free of charge via the Internet at <http://sioc-journal.cn/>.

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