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Crystal-packing modes determine the solid-state ESIPT fluorescence in highly dipolar 2'hydroxychalcones[†]

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This work describes the systematic study of the structure–luminescence relationship of 15 hydroxychalcones directly in the crystal state. Chalcones are easily assembled at the gram scale allowing for efficient variation of their substitution motifs. Our molecule variants combine two modes of fluorescence generation, ESIPT and ICT, both known for their potential to achieve significant quantum yields even with emission in the red to near infrared, a region preferred for technologies as diverse as optoelectronics and chemical sensing. Quantum yields as high as 48% (at 665 nm) and emission wavelengths in the deep red region (710 nm, 5%) were achieved with variants equipped with a strained amino substituent in the donor portion (azetidinyl). Systematic XRD analysis of large monocrystals allowed for the identification of the subtle interplay of several inter- and intra-molecular parameters in achieving such performances, be it intramolecular planarity, non-classical H-bonds, and stacking modes.

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Introduction

Solid-state luminescent materials (quantum dots, polymers, etc.) have been intensively explored over the last decade because of their attractive properties (high quantum vield and photostability) for applications such as chemical sensing, bioimaging and optoelectronics.¹⁻⁶ On the other hand, molecular approaches to solid-state fluorescent materials (organic fluorophores and coordination compounds) have arguably been less frequently investigated due to the well-known quenching effect in the condensed phase, and notably in the crystalline state. π - π interactions or excimer formation in the crystal lattice is largely responsible for the extinction of the luminescence properties exhibited by these molecules in solution.⁷⁻¹¹ In selected cases, this quenching phenomenon is reversed, and has been coined aggregation-induced emission (AIE).7,12 AIE has been largely studied in the amorphous phase upon aggregation from the solution state by increasing the water content. Restriction of intramolecular motions was found to be principally responsible for AIE. Recently, detailed attention to the crystalline phase has identified numerous further parameters

such as hydrogen bonds, electrostatics, orbitals, oscillator strength, *etc.*, whose subtle interplay determines whether solid-state fluorescence is observed. The understanding of this interplay was turned into a theory named solid-state luminescent enhancement (SLE),^{13–16} which emphasizes the multiparametric aspect of any quest to tune solid-state fluorescence.

Consequently, red-shifting the emission wavelength of a fully organic solid-state emitter is a challenge that significantly surpasses that of red-shifting the emission wavelength of solution-phase fluorophores. The usual approach of increasing the size of the π -electron network or enhancing the push-pull character of the substitution pattern often results in fluorescence quenching through detrimental π -stacking interactions or excimer formation once the crystalline state is reached.^{4,17–19} Also, the more red-shifted the fluorophore's emission is, the lower its S1-S0 energy gap is, and the lower its quantum yield is through non-radiative decay.²⁰ Among the red-shifting strategies for solidstate emitters, the four-level energy system, excited-state intramolecular proton transfer (ESIPT) and internal charge transfer (ICT or push/pull), attracted much attention because of their large Stokes shift and efficient long-wavelength emission.²¹⁻²³ Many compounds already reported showed favorable properties with a strong photon emission efficiency.24-26 Combining these two strategies to develop high-performance solid-state fluorophores has thus become a busy research area. In the present study, we elected to work with ESIPT-capable 2'-hydroxychalcones to rationalize their photophysical properties in the solid state. Their easy synthetic assembly allows for the establishment of a variety of substitution



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motifs, and the systematic X-ray diffraction analysis of their crystalline forms might help us to identify intra- and inter-molecular effects thereby promoting high quantum yields and emissions in the red and near-infrared regions.

Experimental methods

2'-Hydroxychalcones are a class of molecules exhibiting ESIPTbased fluorescence as shown in Scheme 1. Ground state chalcone I can reach an excited state (II) upon high-energy photon absorption. Besides the possible non-radiative events, II can then decay through two different radiative pathways: (i) direct de-excitation *via* emission of a photon ($h\nu'$) with a small Stokes shift (typically ≤ 50 nm), regenerating ground state I, or (ii) intramolecular proton transfer (ESIPT) concomitant with an electronic rearrangement generating III (enol form), a lowerenergy excited state. Fluorescence from III ($h\nu''$) is thus observed with a much larger Stokes shift (usually ≥ 100 nm) which leads to the formation of the ground state keto form (IV) that rapidly tautomerizes to regenerate I. With this mechanism in mind, we explored the effects of various substitution patterns on the ESIPT quantum yield and emission wavelength.

Although unsubstituted 2'-hydroxychalcones are reported to show almost no fluorescence in the solid state, placing an electron-donating substituent (EDG) on the phenyl unit leads to intense red fluorescence in this condensed phase.²⁷ Two radiative pathways are therefore possible: the ESIPT mechanism described above, and an internal charge transfer (ICT) promoted by a push-pull effect between the EDG and the keto group (Scheme 2A).²⁸ A few substitution patterns had already been explored for their potential merits for nano-lasers or bioimaging probes.^{26,27,29-33} However, the structure-property relationship remains unclear, even if recent studies tried to rationalize the spectroscopic properties via computational or crystallographic analysis.^{27,30} Indeed, only a few chalcone derivatives with a push-pull substitution have been reported to date. We therefore embarked on the design and synthesis of several new derivatives in order to gain predictive insights into the spectral tuning of this system. Among the fifteen variants prepared in the present work (Scheme 2B), the five derivatives in the 1st series are based on 2'-hydroxyacetophenone (1a-e). In the 2nd group, we investigated the extension of the π system by using a naphthol unit (2a-e), a widely accepted method to obtain polyaromatic near-infrared fluorophores.^{22,34,35} The 3rd group is characterized by the presence of a methoxy substituent

Scheme 1 Excited-state intramolecular proton transfer mechanism for 2'-hydroxychalcones.

Tautomerism



Scheme 2 Synthesis (A) and structural variation (B) of compounds **1a**–e, **2a–e**, and **3a–e**.

(3a-e) in the *para* position relative to the carbonyl group in order to reinforce the ESIPT tautomerization in the excited state, and thus favoring radiative decay and red-shifted emission. For these three groups, we studied the effect of electrondonating substituents on the *p*-position of the phenyl ring, either in the form of a methoxy group (1-2-3e) or a dimethylamino (1–2–3a) group (with Hammett constants σ_p of -0,23and -0.83, respectively).^{36,37} We further examined the influence on crystal packing, and thus fluorescence properties, by varying the substitution motif on the nitrogen to diethylamino (1-2-3b), azetidinyl (1-2-3c) or pyrrolidinyl (1-2-3d). These latter modifications have been chosen to study the impact of the level of molecular planarity on the spectroscopic properties, as their inductive effect is not significantly modulated from one molecule to another. Based on such structural permutations, this study is meant to extract the precious trends that will be key to the design of future near-infrared solid-state emitters.

Compounds **1a–e**, **2a–e**, and **3a–e** (Scheme 2B) were synthesized at the gram scale by simple pyrrolidine-catalyzed aldol condensation in ethanol from the corresponding acetophenones and benzaldehydes, a protocol adapted from a reported method.³⁸ Good to high yields are obtained for our fifteen compounds (57–89%), boding well for further SPR screening in the future. Centimeter-sized crystals of all fifteen chalcones were obtained by liquid/liquid diffusion in CHCl₃/MeOH (Fig. 1), which allowed for the determination of their fluorescence properties in the crystalline state (Fig. S1–S15, ESI[†]).

Results

Excited state keto form

 $\nu'' > \lambda_{h\nu}' > \lambda_{h}$

Ground state

For all crystals, large emission bands were obtained (Fig. 2), which are not only characteristics of solid-state fluorescence, but also of fluorophores operating by an ESIPT mechanism. To our delight, compounds **3c**, **3a** and **2d** showed intense fluorescence in the far-red region ($\Phi = 48\%$, $\lambda_{em} = 665$ nm; $\Phi = 30\%$, $\lambda_{em} = 635$ nm; $\Phi = 13\%$, $\lambda_{em} = 690$ nm, respectively, Table 1), and this occurs despite the fairly small-sized π system, and the



Fig. 1 Pictures of crystals **1a–e**, **2a–e**, and **3a–e** under sunlight (left) and UV irradiation (right).



Fig. 2 Crystal-state fluorescence emission spectra of compounds 1a-d, 2a-e, and 3a-d measured with an integration sphere, with an excitation wavelength of 450 nm (see ESI⁺ for methods). **1e** and **3e** were not fluorescent enough to measure their emission spectrum ($\Phi = 0\%$).

Table 1Crystal-state fluorescence properties of compounds 1a-e, 2a-eand 3a-e, with their intra- and inter-molecular parameters extracted fromDRX analysis. HB = Herringbone, - = compound not fluorescent enoughto measure their emission spectrum

Crystal	Φ (%)	$\lambda_{\max} (nm)$	Arrangement	H/J	d_{ESIPT} (nm)	Torsion (°)
1a	9	675	НВ	J	1.783	10.86
1b	<1	660	HB	J	1.802	24.47
1c	9	670	HB	J	1.783	12.68
1d	14	690	HB	J	1.787	9.95
1e	<1	—	π-Stack	H	1.794	6.89
2a	2	670	π-Stack	J	1.738	8.28
2 b	4	665	HB	J	1.7578	3.34
2 c	5	710	HB	Н	1.724	9.34
2d	13	690	HB	Н	1.729	10.80
2e	<1	630	π-Stack	_	1.777	3.83
3a	30	635	HB	J	1.760	6.13
3b	15	605	HB	J	1.765	4.60
3 c	48	665	HB	J	1.777	3.25
3d	16	680	HB	J	1.774	6.97
3e	<1	_	π-Stack	Η	1.802	26.68

absence of bulky substituents which are often deliberately introduced to avoid dense molecular packing and thus fluorescence quenching. Curiously, all anisole-derived molecules are almost non-emissive (1e, 2e, and 3e). The methoxy group (σ_p = -0,23) might simply be too weak to promote an efficient pushpull mechanism in the excited state.³⁸ In contrast, our candidates with cyclic amine substituents (1, 2, and 3c-d) showed high quantum yields and appreciably red-shifted emissions compared to the ones carrying a dimethyl- or diethylamino group (1, 2, and 3a-b). Particularly, the azetidine-substituted derivatives (1c, 2c, and 3c) stood out for their efficient emission in the far-red region. As the cyclic and open-chain amino substituents exert similar electronic effects (see Hammett constants), these results already hinted at the influence of other intra- or inter-molecular parameters on the fluorescence properties in the crystal lattice. Moreover, extending the π -system by annellation of the acceptor phenol with a 2nd aromatic ring (2ae) did not deliver the desired red-shift of the ESIPT emission band, and a decrease in the quantum yield was observed.

While methoxy substitution of the donor site led to negligible fluorescence, placing a methoxy group at the *para* position of the acceptor portion of the fluorophore led to medium to high quantum yields ($\Phi = 9\%$ to 30% for **1a/3a**, $\Phi < 1\%$ to 15% for **1b/3b**, and $\Phi =$ 9% to 48% for **1c/3c**, Table 1). Unfortunately, this functionalization always blue-shifted the emission wavelength, irrespective of the nature of the amino substituent present. Indeed, the effect of a reduced electron-withdrawing character of the acceptor unit on blueshifting has already been observed in other A–D systems.^{37,39} This was explained with its drastic effect on the internal charge transfer in the excited state, and thus the maximum emission wavelength.

To better understand these experimental results, single-crystal X-ray diffraction (XRD) analysis was carried out for each variant and rationalized in the next section. XRD analysis allowed us to determine for each variant whether it aggregates in J- or H modes and whether it packs in 3D herringbone (HB) or π -stacking modes (Table 1). The obtained data also included intramolecular parameters in the solid state, namely the distance between the phenolic proton and the carbonyl oxygen (d_{ESIPT}) , and the torsion angle between planes formed by the A phenol and D phenyl units (Fig. S16, ESI⁺). As expected, all compounds showed strong innate intramolecular H-bonds between the carbonyl oxygen and the hydroxyl group (1.724-1.802 nm), which is typical of the ESIPT mechanism. Despite this strong H-bonding, which is known to increase the rigidity and the degree of overall molecular planarity (a phenomenon highly favorable for radiative decay), intramolecular torsion angles differed significantly from one chalcone to the other, implying that additional intra- and/or inter-molecular interactions might be responsible for the observed difference in the luminescence properties.

Discussion

It is well known that the mode of crystal packing is paramount to the observed spectroscopic properties in the condensed phase.^{14,27,31} For example, the crystal lattice for **1a** revealed that excimer formation can be avoided even with linear and flat molecules if they are packed as infinite molecular chains with a typical slip-packing structure (or J aggregate, Fig. 3). Conversely, the anisole-based chalcone **1e** also displayed a relatively



Fig. 3 Comparison of J- with H aggregates as found in crystals of **1a** and **1e**, respectively.

planar conformation (torsion angle of 6.89°), but pairs of molecules in the crystal lattice adopted a face-to-face packing mode (or H aggregate), with strong π - π and dipole-dipole interactions; this drastically reduced the quantum yield ($\Phi <$ 1%). Apart from 1e, all variants in series 1 (phenol motifs without any substituent) adopted the desirable J aggregate with an HB 3D arrangement. Intriguingly, all these four variants differed from 1e by displaying on the donor phenyl an N rather than an O substituent. But even within the N series, significant differences in the quantum yield can be detected (from 0 to 14%). For example, 1b showed practically no fluorescence in the solid state. It is also the variant whose torsion angle is substantially greater than that of its congeners (24.47 $^{\circ}$ /1% vs. $9.95^{\circ}/14\%$ for 1d, see Table 1). This correlation has already been observed in a series of stilbene variants.¹³ High degrees of planarity not only promoted the push-pull effect but also the ESIPT mechanism.

In the second series of this study, the phenolic acceptor unit's π -system was enlarged by annellation with an extra benzo ring (Scheme 2B). The resulting naphthol derivatives 2a-e did not show the desired shift of their solid-state fluorescence in the red region (arguably apart from 1d, λ_{max} = 690 nm). The observed quantum yields are also not as easily rationalized from the XRD data. All five compounds showed mostly planar conformations (torsion angles between 3.34° and 10.80°). Yet, only crystalline 2d showed intense fluorescence ($\phi = 13\%$). Strong π - π interactions between naphthol units might be the origin for the insignificant fluorescence of crystalline 2a, despite its favorable J-aggregate slip-packing structure (Fig. S17, ESI[†]). π - π interactions have already been suggested to be the origin for the weak solid-state fluorescence in polyaromatic systems such as porphyrines.^{40–42} The observed packing modes for other variants in this series were more difficult to correlate with the observed spectral properties. Candidates 2b and 2e were almost non-emissive ($\Phi = 4\%$ and $\Phi < 1\%$, respectively), but their crystal lattice exhibited highly favorable HB 3D arrangements with J aggregates (Fig. S18, ESI[†]). On the other hand, 2c and 2d showed a priori unfavorable H aggregation (Fig. S19, ESI[†]); however, while 2c was only weakly fluorescent (ϕ = 5%), 2d was a good contender (ϕ = 13% at 690 nm).



Fig. 4 Degree of planarity and its influence on the quantum yield: amino substituted chalcones.

In the series of 3 variants (Scheme 2B), a *p*-methoxy group was placed on the phenolic acceptor unit. This did not greatly change the molecular conformation and packing modes for amino-substituted chalcones (3a-d), as they were still packed as J aggregates in an HB 3D arrangement (Table 1). However, the XRD analysis revealed that this new presence of an H bond acceptor (O substituent) somehow altered the microenvironment through non-classical H-bonds (Fig. 4). Torsion angles were all small, thus resulting generally in higher quantum yields compared to the unsubstituted series 1, except for 3e. The passage from an N to an O substituent on the phenyl donor unit once again led to a harmful face-to-face packing (or Haggregate), but this time with a far greater torsion angle (26.68° for 3e vs. 6.89° for 1e) ($\Phi < 1\%$, Fig. S20, ESI[†]). Unfortunately, the emission wavelength was strongly affected by this functionalization, as a blue-shift was observed for all compounds compared to variants with no substitution on the phenolic acceptor unit (1a-e). As the ESIPT tautomerization should be reinforced by the presence of such a donor substituent (resulting in a smaller S1-S0 energy gap, and thus red-shifted emission), the observed blue-shift suggested the push/pull fluorescence is dominating the ESIPT mechanism in this series.

Conclusions

To conclude, fifteen ESIPT-active and D-A structured 2'hydroxychalcones were obtained in the crystalline state in this study. This allowed us to determine their suitability as solidstate emitters in the red to near-infrared regions. Candidates with highly promising properties were found, such as 3a and 3c, with red fluorescence and high quantum yields in the crystal state (Φ = 30% and Φ = 48%, respectively). X-ray diffraction analysis allowed us to rationalize the influence of substituents on intra- and inter-molecular interactions on the observed spectral properties. The push/pull character of such chalcones needed to be promoted with para-amino groups in the donor portion as they tend to adopt favorable J aggregates with a Herringbone 3D arrangement, both known to promote radiative decay. On the other hand, replacing such amino substituents with alkoxy donors suppresses the emission. Generally, the degree of planarity correlated well with the emission intensity, as demonstrated in the variant series 1 and 3. Unfortunately, the strategy to extend the π system via benzo

annellation of the phenol unit (2'-hydroxynaphtol series 2) did not deliver the promised benefits in red-shifting and likewise furnished disappointing emission intensities. As another deviation from the rule, the π stacking between polyaromatic cycles in crystalline **3a** did not cause notable quenching of its fluorescence. As XRD analysis could not explain all observed trends, molecular modeling might contribute to rationalize the photophysical properties of such compounds. In conclusion, molecular conformation and crystal packing of 2'-hydroxychalcone derivatives could be strongly tuned by simple chemical modifications. We hope that this study will help in future efforts to rationally design solid-state molecular fluorophores.

Conflicts of interest

The authors declare no competing financial interest.

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