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A series of schiff base derivatives with salicylaldehyde moieties were designed and synthesized. This materials exhibit typical AIE properties and high fluorescence quantum yields in an aggregated state based on excited-state intramolecular proton transfer (ESIPT) mechanism. Then, the effect of substituted group types and positions on the fluorescence of these novel compounds was investigated through fluorescence spectra analysis and theoretical calculations. And it was found that the salicyaldehyde derives can be used as potential pH and Zn²⁺ sensing.

Introduction

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Organic light-emitting materials have attracted significant focus in the field of optoelectronic devices and fluorescent biosensors by virtue of their structural varieties, rich colours, good tunability, flexible molecular preparation and excellent physical and chemical properties.¹ However, one key problem associated with real applications is aggregation-caused quenching (ACQ) effect. Most traditional organic luminophors contain planar π -conjugated aromatic rings, which can increase the chances for molecular aggregation to form excimers and exciplexes in the solid state or in concentrated solutions. They show strong fluorescence in dilute solution, but become weakly emissive in the aggregate and film states due to the nonradiative deactivation of the excited state of excimers and exciplexes.² The ACQ effect heavily limits their practical applications of luminescent materials in organic lightemitting diodes and fluorescent biosensors, which require materials to be films or high concentration.³

To solve the thorny ACQ problem of many widely used most luminescence, an aggregation-induced emission (AIE) phenomenon was reported by Tang and colleagues, which is exactly opposite to the ACQ effect.⁴ It was found that the restriction of intramolecular rotations (RIR) process is responsible for the AIE effect: the non-luminescence of 1methyl-1,2,3,4,5-pentaphenylsilole molecules in solution is attributed to the active intramolecular rotations of phenyl rings on its periphery, whereas the strong emission in the aggregate state is caused by the restriction of intramolecular rotations of these benzene rings. On account of their unique



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In the past ten years, AIE mechanisms involving restriction of intramolecular rotation (RIR), twisted intramolecular charge transfer (TICT), cis-trans isomerisation, excited-state intramolecular proton transfer (ESIPT) have been reported, etc.⁸ Among these merging AIE emission mechanisms, ESIPTbased luminophors generally display excellent signal performance on the account of dual emission, large Stokes shifts (~200nm) as well as good photostability, and spectral sensitivity to the surrounding medium.⁹ A series of salicylidene-based materials with AIE properties have been reported. Nevertheless, they exhibit the shortcomings of less fluorescence quantum efficiencies and short fluorescence lifetime in the solid state.¹⁰

Herein, we synthesized a new series of Schiff-base compounds (1-3) with salicylaldehyde moieties as shown in scheme 1. They exhibit typical AIE properties with various fluorescence emissions from green to red, high fluorescence quantum yield and long fluorescence lifetime in an aggregated state. Meanwhile, the Schiff-bases show unique pH-dependent optical properties in acidic condition and Zn²⁺ responsive fluorescence, which indicated their potential application prospects in chemosensors for pH and Zn²⁺ sensing



N(CH₃)₂ N(CH₃)₂ NO₂ $R_2 =$

Scheme 1 Synthesis of compound 1-3.



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Results and discussion

AIE characteristics

The fluorescence characteristics of 1-3 were investigated in EtOH/water mixtures (water fraction (f_w) from 0% to 90%, v/v) buffered by 10 mmol•L⁻¹ Tris-HCl at pH 7.4. As shown in Fig. 1B, the fluorescence emission of 1 was weak in EtOH and increased slowly until f_w reached 70%. Afterwards, the fluorescence emission rose swiftly, which increased by 36-fold from the EtOH solution to 90% aqueous mixture. 2 and 3 behaved similarly (Fig. 1C, 1D): they exhibited weak fluorescence emission in EtOH solution and in EtOH/water mixtures with f_w lower than 40% and 50%, respectively. When $f_{\rm w}$ were beyond 40% and 50%, their fluorescence emissions enhanced remarkably, which increased by 23-fold and 20-fold from the EtOH solution to 90% aqueous mixture, respectively. Interestingly, 1-3 exhibited different emissions from green to red: the corresponding maximum emission wavelengths are 542 nm, 572 nm and 632 nm, respectively. These might be due to the intense electronegativity of substituent groups, which influenced the electronic cloud distribution of the molecule.

To further investigate the reasons that account for the difference in optical properties of 1-3 in aggregated states, we performed density functional theory (DFT) calculations to gain a better understanding of the geometric and electronics structure. The DFT calculations were carried out with Gaussian 03 program. The geometries were optimized with the B3LYP hybrid density functional DFT and the basis set used was 6-31G*. Fig. 2 displays the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) diagrams of compound 1-3, and the calculated HOMO and LUMO energies and energy gaps (E_g) are corresponding listed. The molecular orbital amplitudes of 1 and 2 suggested that the HOMOs are generally distributed on the 4-N,N-dimethylaminoaniline group, while the LUMOs distributed more on the 4diethylaminosalicylaldehyde group and 5-fluorosalicylaldehyde group, respectively. The molecular orbital amplitudes of 3 revealed that the HOMO is generally distributed on the 4diethylaminosalicylaldehyde group, while the LUMO distributed mostly on the 4-nitroaniline group. The strongly electron-withdrawing substituents -F and -NO2 contribute to the more separated electronic cloud of HOMOs and LUMOs, which decrease the HOMO energies (1: -0.92 eV, 2: -1.57 eV, 3: -2.45eV) and LUMO energies (1: -4.46 eV, 2: -4.92 eV, 3: -5.63 eV) as well as Eg results (1: 3.54 eV, 2: 3.35eV, 3: 3.18 eV) of the compound $\mbox{1-3}.$ The E_g is correlated with the wavelength of fluorescence emission: small E_g enables the electron transition, easily resulting in a red shift of maximum emissions from 542 to 632 nm.

The changes of fluorescence spectra in EtOH/water mixtures indicated that **1-3** showed typical AIE characteristics. We also studied the emission behaviors of **1-3** in the solid state. Fluorescent spectra and photographs under UV light 365 nm are given in Fig. 3. The fluorescence quantum yields (\mathcal{O}_F) of the **1-3** solid were 27.8%, 55.7% and 68.1% measured by using an integrating sphere, respectively. The results of \mathcal{O}_F are superior to the reported AIE molecules based on salicylaldehyde Schiff-



Fig. 1 Fluorescence photographs (A) and fluorescence emission spectra of (B)**1**, (C)**2**, (D)**3** in EtOH/water mixtures with different f_w . Conditions: the concentrations of **1-3** are 30.0 μ mol·L⁻¹. The excitation wavelength is 408 nm, 392 nm, 445 nm, respectively. The photographs were taken under irradiation of 365 nm UV light.



Fig. 2 Frontier molecular orbital amplitude plots and energy levels of the HOMOs and the LUMOs of **1-3**.



Fig. 3 The emission spectra (3A) of **1-3** powder Excited at 408 nm, 392 nm, 445 nm, respectively. And the decay dynamics (3B), fluorescence photographs (3C) of the corresponding neat film under irradiation with UV light at 365 nm.

base (1.2%-17.0%).¹¹ The increasing trend of these three compounds in fluorescence quantum yields can be well explained by the spatial conformation. According to the DFT calculations, it was found that molecules 1-3 adapt twisted conformations both the aromatic rings, and their twist angles are 11.08°, 10.39°, 9.86°, respectively. It is generally acknowledged that the planar conformations are beneficial to ESIPT because of an increasing ESIPT barrier correlating with the increasing twist angle. On the basis of molecular structure, it has a twisted structure at the energetic minimum of the ground state; while a planar structure are more stable in the excited state. Hence, for the planar molecules, the ESIPT process could occur directly.¹² For molecule 3, it has the minimum twisted conformation, which results in the easiest ESIPT, thus explaining the highest fluorescence quantum yield. The PL decay dynamics of the compounds 1-3 were studied by a time-resolved technique (Fig. 3B and insert table). In the neat film, the decay curves of 1-3 are good fitted by a doubleexponential function (Chi-squared distribution of goodness of fit is 1.04, 1.05, 1.13, respectively), indicating that two relaxation pathways are involved in the decay process. For compound 1-3 film, the quick decay lifetime is 0.11µs (14.26%), 0.12µs (14.78%) and 0.12µs (16.72%), respectively, while long decay lifetime is 0.69µs (85.74%), 0.74µs (85.22%) and 0.83µs (83.28%), respectively. The longer lifetime for the excited states account for the higher emission efficiency, which is in accordance with the quantum yield results.

ESIPT mechanisms of AIE

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After studying the optical properties of compound 1-3 in EtOH/water mixtures, we further investigated the mechanisms of AIE. ESIPT is phototautomerization with enol form (E) be changed to keto form (K) upon photoexcitation, by migration of a proton to the neighboring electronegative atom via intramolecular hydrogen bonding (Fig. 4). On relaxation of the keto form to the ground state, the enol form is recovered by reverse proton transfer. The presence of intramolecular hydrogen bonding between the acidic proton and basic moiety is crucial. Acidic protons are usually -OH and -NH₂ and basic centers are =N- and carbonyl oxygen (C=O). Salicylaldehyde Schiff-base derivatives 1-3 are ESIPT dyes. In compound 1 dual emissions from the enol and keto forms were observed in EtOH/water mixture (Fig. 2B), the enol tautomer gives emission at shorter wavelength 515 nm and the keto tautomer gives emission at longer wavelength 542 nm. A similar trend was observed for compound 2. It also exhibits ESIPT in EtOHwater mixture, as evidenced from the strong enol emission at 552 nm and strong keto emission at 595 nm. For compound 3, the fluorescence spectra show unimodal broad curve, which possibly attribute to the doublication of enol emission and keto emission because of its two channel decay of fluorescence lifetime as well as compound 2 and 3. The ESIPT chromophores 1-3 obtain high quantum efficiencies and large stokes by harvesting the ESIPT process.

pH-dependent optical properties

The pH responses to fluorescence properties were investigated for **1-3** (30.0 μ mol·L⁻¹) in aqueous solution ($f_w = 90\%$) buffered by Tris-HCl at pH 3.0-7.0. All types of fluorescence measurements were monitored parallelly 3 times at same conditions. As shown in Fig. 5B-D, **1-3** exhibited strong

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J. Name., 2013, **00**, 1-3 | **3**

fluorescence emission when pH > 5.5 and different pHdependent fluorescence response in the mange og / pbk & 595 (5) Three compound showed titration jump at pH around 4.0, 3.5 and 3.5, respectively. Fig. 5A shows a corresponding fluorescence photo at different pH values. Herein, 1-3 exhibited different stability under acidic conditions, which is due to the negative inductive effect of substituent groups in salicylaldehyde moieties and phenylamine moieties. Normally, the hydrolysis of Schiff-base in acid solution is ascribed to an electrophilic addition reaction. The electron density of the C=N bond dominates the stability of Schiff-base. For compound 1, there are two electron-donating groups diethylamino group in the salicylaldehyde moiety and dimethylamino in the phenylamine moiety, so the C=N double bond in 1 has the lowest stability. Fluorine in the salicylaldehyde moiety of compound 2 and nitro in the phenylamine moiety of compound **3** are strongly electron-withdrawing group and have high electronegativity, which reduces the electron density of the C=N bond in Schiff-base. Thus, higher hydrogen ion concentration was needed to hydrolyze the C=N bond in 2 and **3**. The titration jump of **2** and **3** occurred at a lower pH (3.5) than that of 1 (4.0). The possible mechanism of pH response of 1-3 as shown in Fig. 6.



Fig. 4 The photophysical cycle of **1-3** for illustration of ESIPT process.



Fig. 5 Fluorescence emission peak intensities (B:1, C:2, D:3)

and fluorescence photographs (A) of 1-3 in 90% water/EtOH

(v/v) at different pH values. Conditions: the concentrations of

1-3 are 30 μ mol·L⁻¹. The photographs were taken under an

irradiation of 365 nm UV light.



Scheme 2 The possible mechanism of pH response of 3.

In order to further investigate the mechanism of pHdependent optical properties, the comparative trial of ESI-MS spectra of compound **3** and treatment **3** with hydrochloric acid were tested. In pure methanol solution, only the molecular ion peaks of **3** were obtained at m/z 314.1 (Fig.S9, ESI). After treatment with hydrochloric acid, different molecular ion peaks of 4-diethylaminosalicylaldehyde at m/z 194.1 ($[M+H]^+$) and 4-nitroaniline at m/z 180.1 ($[M+N_a^++H_2O]$) could be clearly observed (Fig. S10, ESI). The results suggested that **3** undergo decomposition reaction with acid (scheme2).

Selective response of 2 to Zn²⁺

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It is well known that Zn²⁺ plays an important role in a wide range of biochemical processes. The disturbance of Zn²⁺ metabolism is closely associated with serious diseases such as Parkinson's disease, Alzheimer's disease, epilepsy, hypoxiaischemia, dysgenesis and amyotrophic lateral sclerosis. In addition, excessive Zn²⁺ is also harmful to the environment.¹³ Therefore, it is important to develop an efficient and sensitive method to detect Zn²⁺. In recent years, although a series of fluorescent chemosensors for Zn²⁺ have so far been reported, a lot of them easily disturbed by ACQ effect and possessed only poor detection limits.¹⁴ However, the ratiometric chemosensor based on AIE-active molecule would have the unique advantages over conventional chemosensor: Firstly, increasing its fluorescence color responses by large Stokes shift (approximately 115 nm); Secondly, improving its ratiometric sensitivity. detection The fluorescence chemosensor could reduce the interference of background and show better linear relation; And thirdly, reducing ACQ effects of traditional fluorophores. The fluorescence of AIE mechanism can be easily adjusted by the analyte through influencing the aggregation behaviour of fluorophores.¹⁵

For compound 2, it shows excellent selectivity for Zn^{2+} in EtOH/H₂O (1/2, v/v, pH 7.4) Tris-HCl buffer solution. As shown in Fig. 6 compound **2** (10.0 μ M) has a signigcant yellow fluorescence. Upon the addition of Zn^{2+} (1.0 eq.), the maximum emission peak of **2** blue shift from 552 nm to 460 nm, while other tested metal ions lead to hardly any noticeable spectral changes. The results of competition experiments indicate that the fluorescence intensity of **2** in the presence of Zn^{2+} (1.0 equiv.) is unaffected by the addition of most competing metal ions. However, Cu²⁺ can prevent emission peak from blue shift after they were added to **2** (Fig. 7).

The gradual changes of the fluorescence spectra of **2** (10.0 μ M) upon addition of Zn²⁺ (0 to 18.0 μ M) are shown in Fig. 8, obviously, compound **2** can be utilized as an ratiometric fluorescence sensor for Zn²⁺ (Fig. 8a). Accordingly, the data analysis revealed an excellent linear relationship: y = 2.96x-0.29 (R²=0.990) between the ratiometric fluorescence



Fig. 6 Fluorescence spectra of compound **2** (10.0 μ M, λ_{ex} = 361 nm) with the addition of various metal ions (10.0 μ M) in EtOH/H₂O (1/2, v/v, pH 7.4) Tris-HCl buffer solution.



Fig. 7 Fluorescence spectra of **2** (10.0 μ M) with Zn²⁺ in the presence of various metal ions (10.0 μ M) 1:Cu²⁺, 2:Hg²⁺, 3:Pb²⁺, 4:Na⁺, 5:K⁺, 6: Ba²⁺, 7:Fe³⁺, 8:Cr³⁺, 9:Al³⁺, 10:Ag⁺, 11:Mg²⁺, 12:Co²⁺, 13:Ni²⁺, 14:Ca²⁺, 15: **2**.



Fig. 8 (a) Fluorescence titration spectra of 2 in the presence of different concentrations of Zn^{2+} (0–1.8 equiv.) in EtOH/H₂O (1/2, v/v, pH 7.4) Tris-HCl buffer solution. (b)normalized response of fluorescence signal of **2** in the presence of different concentrations of Zn^{2+} (1.0-10.0 μ M). (c) Job's plot of **2** and Zn^{2+} when the total concentration of **2** and Zn^{2+} was 10.0 μ M. Excitation wavelength $\lambda_{ex} = 361$ nm.

Journal Name

 (F_{460}/F_{552}) and the Zn^{2+} concentration (1.0-10.0 μM). The detection limit of **2** were calculated to be $0.26\mu M$ (Fig. 8b) by DL=3\sigma/s (σ =2.57%). The results of the Job's plot experiment show a 2 : 1 stoichiometry complexation between **2** and Zn^{2+} (Fig. 8c). The plausible binding mode of **2** to Zn^{2+} was shown in scheme 3. Although compound **1** and **3** are also response for Zn^{2+} , and show blue shift of emission, their fluorescence quenched significantly (Fig. S12 and Fig. S13). Therefore, the detection effects of **1** and **3** for Zn^{2+} are poorer than that of **2**.



Scheme 3 Proposed binding mode of 2 with Zn²⁺.

Conclusions

Three new Schiff base derivatives possessing AIE characteristics with various fluorescence emissions from green to red and high fluorescence quantum yields in the aggregated state were synthesized using a facile and efficient method. The different substituent groups in the salicylaldehyde moiety(R_1) and in phenyl group(R_2) produce the difference of optical properties. These multiple-colour AIE molecules have a potential application of novel pH and Zn²⁺ chemosensors.

Experimental

Chemicals

Analytical grade absolute ethanol, dimethyl formamide deionized water (distilled) were used throughout the experiment as solvents. All materials for synthesis were purchased from Aladdin Industrial Corporation (China) and used without further purification.

Apparatus

¹H NMR spectrum was run on a Varian Mercury-Plus 300 NMR spectrometer using TMS as the internal standard (Varian, San Diego). Mass spectrum was recorded with a VG ZAB-HS double focusing mass spectrometer (Thermo Onix Ltd, UK). Fluorescence spectra, fluorescence lifetime and quantum efficiency were measured with a Fluorolog 3-TSCPC and integrating sphere accessory (Horiba Jobin Yvon Inc. France). Infrared Spectroscopy was measured using Nicolet 5700 (Thermo Electron Scientific Instruments Corp., America). **Syntheses**

4-N,N-dimethylaminoaniline-4-diethylaminosalicylaldehyde Schiffbase (1)

4-diethylaminosalicylaldehyde (0.19g, 1.0 mmol) and 4-N,N-dimethylaminoaniline (0.14g, 1.0 mmol) were dissolved in anhydrous ethanol (10mL). The reaction mixture was stirred for 10h at 60° C and then the mixture was cooled to room

temperature. The precipitate was filtered off, washed with cold ethanol one time and dried in vacuum to give the desired product as a green solid in a 69% yield. m.p. 162.1-164.5 °C, ¹H NMR (300 MHz, DMSO-d₆, δ ppm): 1.10 (t, 6H), 2.90 (s, 6H), 3.35 (q, 4H), 6.03 (d, 1H), 6.25 (d, 1H), 6.72 (d, 2H), 7.19 (d, 2H), 7.23 (d, 1H), 8.61 (s, 1H), 13.90 (s, 1H). FT-IR (KBr, cm⁻¹): 3420 (ν OH), 2969, 2890 (ν CH₃), 1620(ν C=N), 1352 (ν C-N), 1524, 818 (ν p-phenyl), 1213 (ν Ar–OH), 1124 (ν C-C). MS m/z: 312.2 ([M+H]⁺). M⁺ calculated 311.2.

4-N,N-dimethylaminoaniline-5-fluorosalicylaldehyde Schiff-base (2)

5-fluorosalicylaldehyde (0.14g, 1.0 mmol) and 4-N,N-dimethylaminoaniline (0.14g, 1.0 mmol) were dissolved in anhydrous ethanol (10mL). The reaction mixture was stirred for 8h at 60 °C and then the mixture was cooled to room temperature. The precipitate was filtered off, washed with cold ethanol one time and dried in vacuum to give the desired product as a yellow solid in a 78% yield. m.p. 155.3-157.9 °C, ¹H NMR (300 MHz, DMSO-d₆, δ ppm): 2.94 (S, 6H), 6.76 (d, 2H), 6.89 (d, 1H), 7.14 (d, 1H),7.33 (d, 2H), 7.39 (d, 1H), 8.87 (s, 1H), 13.30 (s, 1H). FT-IR (KBr, cm⁻¹): 3415 (v OH), 2896,2814 (v CH₃), 1615(v C=N), 1363 (v C-N), 1524, 1490, 818 (v p–phenyl), 1269 (v Ar–OH), 1207 (v C–F), 1140 (v C-C). MS m/z: 259.1 ([M+H]⁺). M⁺ calculated 258.1.

4- nitroaniline-4-diethylaminosalicylaldehyde Schiff-base (3).

4-diethylaminosalicylaldehyde (0.19g, 1.0 mmol) and 4nitroaniline (0.14g, 1.0 mmol) were dissolved in anhydrous ethanol (10mL). The reaction mixture was stirred for 6h at 60 °C and then the mixture was cooled to room temperature. The precipitate was filtered off, washed with cold ethanol one time and dried in vacuum to give the desired product as a red solid in a 65% yield. m.p. 165.9-166.7 °C, ¹H NMR (300 MHz, DMSOd₆, δ ppm): 1.10 (t, 6H), 3.37 (q, 4H), 6.07 (d, 1H), 6.36 (d, 1H), 7.36(d, 1H),7.48 (d, 2H), 8.22 (d, 2H), 8.78 (s, 1H), 13.16 (s, 1H). FT-IR (KBr, cm⁻¹): 3435 (υ OH), 2980 (υ CH₃), 1630(υ C=N), 1569 (υ Ar-NO₂),1325 (υ C-N), 1514, 818 (υ p-phenyl), 1235 (υ Ar-OH), 1141 (υ C-C). MS m/z: 314.1 ([M+H]⁺). M⁺ calculated 313.1.

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A series salicyaldehyde derives with AIE properties were designed and synthesized, which can be used as potential pH and Zn^{2+} sensing.

