Chinese Chemical Letters 34 (2023) 107457

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Visible light responsive spiropyran derivatives based on dynamic coordination bonds

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ARTICLE INFO

Article history:
Received 26 March 2022
Revised 17 April 2022
Accepted 22 April 2022
Available online 27 April 2022

Keywords:
Negative photochromic
Spiropyran
Visible light responsive
Dynamic coordination bonds
Design guidelines

ABSTRACT

Spiropyrans (SPs) are a well-known class of photochromic compounds and have found widespread application due to their unique properties. However, for many conventional SPs, high energy ultraviolet (UV) light is commonly essential to drive photoisomerization, leading to poor fatigue resistance. Moreover, the practical application of spiropyrans is hindered by their fast fading speed due to the instability of closed forms (SP) or open forms (MC). Herein, we disclose a novel strategy to address these challenges through introducing both electron-donating substituents to stabilize the SP and dynamic coordination bonds to stabilize the MC. The resulting new spiropyrans complexes exhibit negative photochromic properties, with fast visible light response, good stability of both SP and MC, and significantly improved fatigue resistance.

Photochromatic spiropyran (SPs) derivatives have attracted intense interest for their potential applications in various fields, such as fluorescence imaging probes [1–6], biosystems [7–10], chemical sensors [11–14], smart windows [15,16], photoacids [17–19], security printing [20,21] and anti-counterfeiting materials [22–24]. However, the practical application of SPs is hindered by two critical challenges: (1) The open forms (or MC) are unstable and will turn back to the original closed forms (or SP) quickly even in the dark, resulting in fast color fading [25]; (2) High energy ultraviolet (UV) light is commonly used to initiate the switching process of SPs [26–28], which will destroy the π-conjugated structures of MC and leads to poor fatigue resistance [29,30]. It is highly desirable to develop SPs with slow fading speed and enhanced fatigue resistance.

Typically, the fading speed of MC was mainly retarded by improving their stability. It is known that the introduction of electron-withdrawing groups at 6-position on the chromene portion to the spiropyran molecule will significantly improve the UV responsiveness and lead to the stabilization of the open form [31–33]. Introducing metal ions into spiropyrans can also stabilize the merocyanine structure due to the formation of stable coordination complexes [34–37]. However, the currently reported metal ion-interacting spiropyran derivatives still exhibit many demerits (Table S1 in Supporting information). On the one hand, stabilization of the open form will generally destabilize the closed forms, sometimes will even change the photochromic direction. In some cases, the SP forms rapidly equilibrated back to the MC form after removing the visible light source (Table S1) [35]. In this situation, the MC states of the spiropyran compounds will be observed predominantly, and it is difficult for them to be switched to the SP states on demand. For some metal ion-complexed spiropyrans, their MC forms are too stable after chelating with metal ions, resulting in their inability to be recovered, or must be recovered with the addition of chemical stimuli, such as EDTA or protons (Table S1) [38–43]. Nevertheless, a reversible transformation through a chemical-free and safe method is the basic requirement for many applications. So far, few reports have focused on simultaneously balancing the stability of MC and SP states, especially by designing electron-donating substituents to improve the stability of the closed form [33].

On the other hand, for many metal ion-interacting spiropyran derivatives, high-energy UV light is commonly necessary to drive the photoisomerization process, or they can chelate with certain metal ions only when they are transformed into an "active" state with the assistance of ultraviolet light (Table S1) [44–50]. However, for most applications (especially for applications in biological systems), UV light is not suitable, as high-energy UV light usually damages cells, trigger unwanted reactions (such as apoptosis), and have poor selectivity due to non-selective absorption by common chromophore [51,52]. In this scenario, developing visible light responsive chromophores would be attractive because visible light is non-invasive, environmentally harmless, dominant in

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https://doi.org/10.1016/j.cclet.2022.04.055
1001-8417/

the solar spectrum, has a wide wavelength window, and can minimize degradation and improve fatigue resistance [53–55]. Moreover, visible light is spatially and temporally controllable which can be readily tuned and focused to provide "on-demand" access and achieve selective excitation for broad applications [54].

Some strategies have been developed to redshift the excitation wavelength of photochromic molecules to the visible light range, including direct visible-light excitation of extended π conjugation, push-pull systems and indirect visible-light excitation of a suitable sensitizer [56–58]. Designing negative photochromic compounds whose stable form is red-shifted relative to the unstable form, is also an effective strategy to obtain photo-switching materials driven by visible light [59,60]. Nonetheless, few studies have applied these strategies to spiropyrans in the literature [61,62]. Moreover, spiropyrans which show both reduced color fading and enhanced visible light photoactivity have not been reported so far.

Herein, we report a novel strategy to design visible light-responsive spiropyrans. In contrast to previous studies which commonly install electron-withdrawing nitro group to increase the stability of MC, here we grafted electron-donating groups at the 6-position on the chromene portion to stabilize the SP forms. Meanwhile, we introduced an ester group at carbon C_8 (ortho to oxygen) at the pyran half to form a bidentate ligand to enhance the stability of MC forms. Zinc(II) ion was selected as metal center because zinc(II) has a d^{10} electron configuration which is favorable for the formation of dynamic coordination bonds [63–65]. The introduction of ester groups with weak coordination ability and labile metal ions allows the metal complexes of the MC forms to achieve reverse photochromism without additional chemical stimulation. As a result, the new spiropyrans are UV light inactive but can transform to open structures though addition of metal ions. The resulting metal complexes of the new spiropyrans exhibit negative photochromic properties, fast visible light response, good stability of both SP and MC-forms and significantly improved fatigue resistance.

The synthetic routes are shown in Schemes S1-S3 (Supporting information). The final products were characterized by NMR, FT-IR and HR-MS (Figs. S1–S17 in Supporting information). We firstly investigated their UV response. As shown in Fig. S18 and Table S2 (Supporting information), the new SPs (OIC and MIC, Fig. 1) exhibits two obvious absorption peaks at 280–290 nm and 330–350 nm, respectively, which can be attributed to the $\pi-\pi^*$ electronic transitions in the indolene (250–290 nm) and the chromene moieties (300–350 nm) [66–68]. The color and absorption spectra show no change after UV light irradiation (365 nm) for 3 min (Figs. 2a and b left, Fig. S18 in Supporting information), indicating that these SPs are UV light inactive. The effect of different wavelengths of UV light was also verified. The results showed that the MIC solution did not respond to UV light at 254 nm either (Fig. S19 in Supporting information). In contrast, for the traditional SPs (NSP and NSIC, Fig. 1) the color of the solutions changed from colorless to purple and new absorbance peaks were observed around 543 nm after UV exposure (Figs. S20a, c and d in Supporting information), due to the formation of MC forms [69].

Surprisingly, the SP to MC switching process of our new SPs compounds can be initiated by adding metal ions. As shown in Fig. 2a, the color of MIC solution changed gradually from colorless to red after adding Zn^{2+} ions. A new absorption peak characteristic for $\pi-\pi^*$ transition of MC form was observed at 527 nm (Fig. 2c and Table S2) [70]. The absorption intensity at 527 nm increased continually with the increase of Zn^{2+} ions, indicating that more and more MIC compounds were transformed into MC form (Fig. 2d). A saturation of the absorbance was not observed due to the small equilibrium constant of the coordination reaction. Similarly, OIC exhibit apparent color change from colorless to purple and absorbance change upon addition of Zn^{2+} ions (Figs. 2b–d and Table S2). Noteworthily, adding zinc ion into the solutions of NSP and NSIC did not cause any color or absorbance change (Figs. S20b–d in Supporting information), indicating no coordination bonds were formed in these cases.

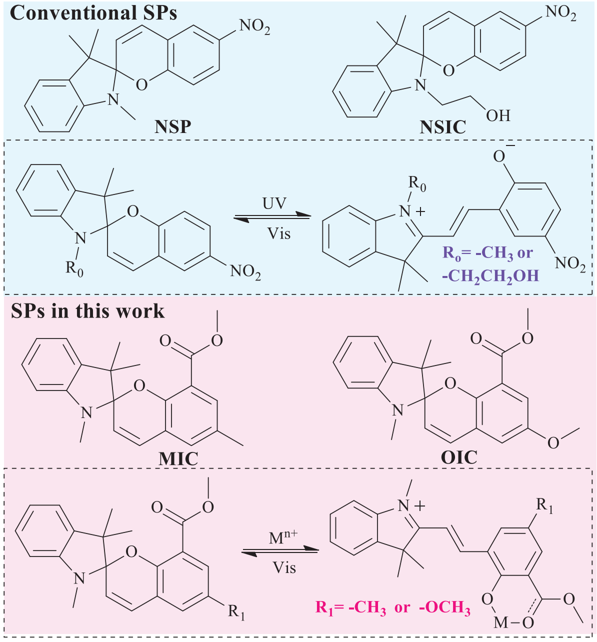

Fig. 1. Structures and photochromic process of conventional spiropyrans in the literature and new spiropyrans in this work.

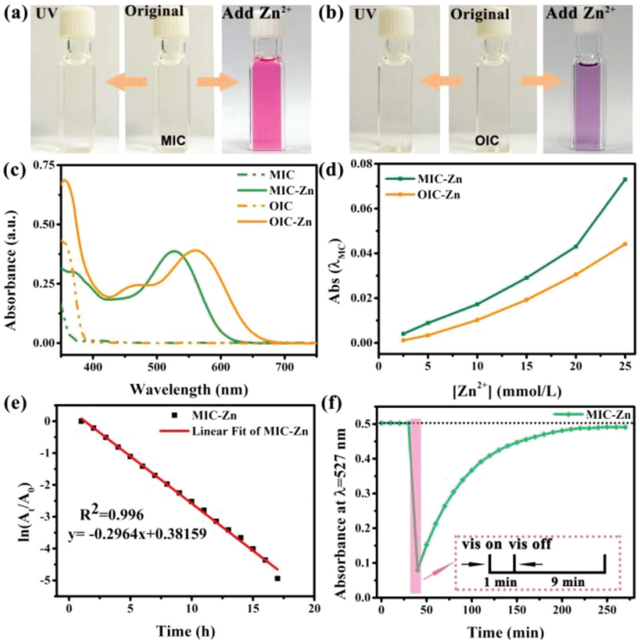

Fig. 2. The photographs of (a) MIC and (b) OIC, (middle) original SPs solution (50 µmol/L) and (left) after UV irradiation, (right) after adding Zn^{2+} ions (90 mmol/L, methanol, 293 K), respectively. (c) Absorption spectra of SPs (50 µmol/L, THF, 293 K) before and after adding zinc ions (90 mmol/L). (d) Absorbance of SPs solution (50 µmol/L) at λ_{MC} with different Zn^{2+} concentration. λ_{MC} represents the absorbance wavelength of SPs in their MC form (at 527 nm for MIC and 561 nm for OIC). (e) Fading kinetic curve of MIC-Zn (50 µmol/L). (f) Absorbance evolution of MIC-Zn *versus* time before and after visible light irradiation.

The formation of MC-Zn complexes (marked as MIC-Zn and OIC-Zn, respectively) is a gradual process, as they need relatively long time (150 min for MIC-Zn and 10 min for OIC-Zn) to reach maximum absorption (Fig. S21 in Supporting information). To investigate whether UV-light can accelerate the isomerization process, a comparison experiment was performed by alternately placing the Zn^{2+}-containing MIC solution under UV light and without UV light in the dark for 30 min to measure the maximum absorption peak of its open form (Fig. S22 in Supporting information). The results indicated that UV light could not trigger or accelerate the isomerization process. In contrast, it inhibits the formation of MIC-Zn. This is because that the electron-donating groups at the 6-position on the chromene portion enhanced the stability of the SP form. Therefore, the new spiropyrans are inactive to ultraviolet light due to the inability to induce cleavage of C_{SP}-O bond like conventional spiropyrans [10,71,72]. On the contrary, UV light breaks metal–ligand bonds that formed already in the MC-complexes [73–75]. The dissociated spiropyran can be quickly recovered to the SP state, which leads to a significant decrease in the absorption peak of the MC form (Fig. S22). This conclusion is consistent with the UV resistance properties described above.

The zinc complexes of MIC in MC form can be transformed back to the SP forms under visible light. As shown in Video S1 (Supporting information), after irradiation of 8 W LED white light torch for 5 s, MIC-Zn turned to colorless. Interestingly, such transition process can also be initiated under irradiation with AM 1.5 sunlight within 60 s (Fig. S23a in Supporting information). What is more, under outdoors insolation sunlight, the MIC-Zn solution will fade quickly (Fig. S23c and Video S2 in Supporting information). The purple solutions of zinc complexes of OIC (*i.e.*, OIC-Zn) show the similar property (Fig. S23b and Video S3 in Supporting information). Such negative photochromic behavior, especially under visible light with mild intensity, has been rarely reported before. It is worth mentioning that MIC-Zn complexes are also reversible under UV irradiation at different wavelengths accompanied with a decrease in absorption intensity and a color (Fig. S24 in Supporting information). This is ascribed to the anti-UV properties of the new spiropyrans and stability of its SP state, which has rarely been reported.

The photochromic processes of our new spiropyrans do not rely on UV light, which should be of great benefit for the improvement of the fatigue resistance. As shown in Fig. S25 (Supporting information), after 25 cycles, spiropyrans with methyl ester (OIC and MIC) presented slight degradation. Compared to the spiropyrans with nitro group (NSP or NSIC) (Fig. S26 in Supporting information), its anti-fatigue performance is correspondingly improved. The fading kinetic of MIC-Zn was conducted by tracking the absorption spectra of the MIC-Zn solution on certain interval in dark (Fig. 2e). The results indicate that the merocyanine form of MIC-Zn was stable with a quite small rate constant ($k = 0.2964$ h^{-1} according to first-order decay process [33,76,77], $A_t = A_0\exp(-kt)$) and a relatively long half-life of 2.34 h. We further found that, compared with MIC-Zn, OIC-Zn has a slower decay rate ($k = 0.1033$ h^{-1}) and a longer life of 6.71 h (Fig. S27 in Supporting information). They can even be stored at ambient conditions without being protected from natural light for over 5 h while showing insignificant color fading (Fig. S28 in Supporting information), indicating that the merocyanine formed after the addition of zinc ions is relatively stable. Interestingly, the bleached solutions also have good stability. After white light irradiation, it takes about 3.3 h for MIC-Zn (Fig. 2f) and 20 min for OIC-Zn (Fig. S29 in Supporting information) to return to MC from SP. The recovery time of OIC-Zn was found in a much shorter time compared to MIC-Zn. It can be attributed to the stronger electron donating ability of methoxy than methyl [78], which improves the rate of ring opening by increasing the electron density of oxygen [79] and enhances the coordination ability of the oxygen atom bound to the spirocarbon atom [80]. Nevertheless, these recovering times are significantly longer than previously reported works, especially materials based on coordination reactions [33–35,81,82].

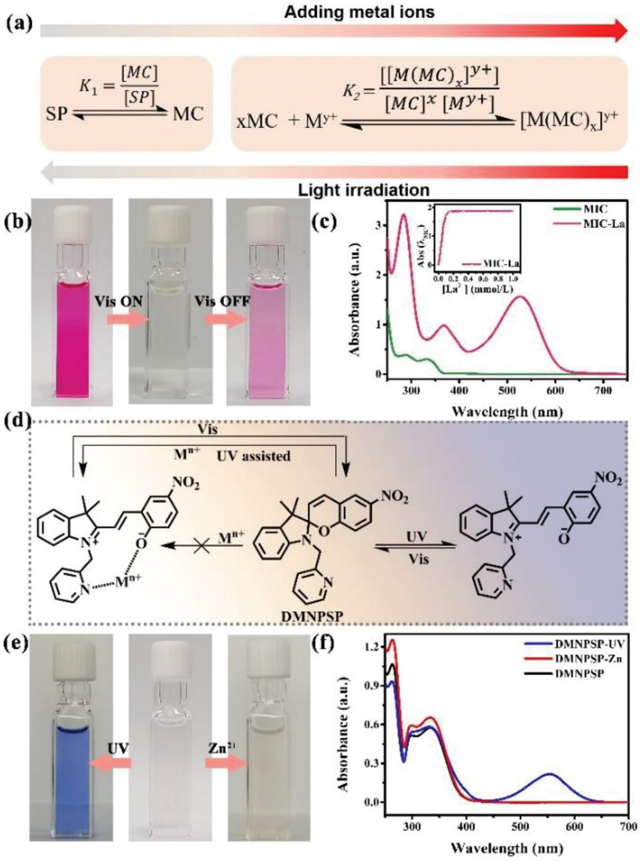

Fig. 3. (a) The equilibrium constant (K_1 or K_2) between the SP and MC or MC and $M[(MC)_X]^{y+}$. (b) Photographs of MIC (50 µmol/L, THF, 293 K) solution with La^{3+} ions (90 mmol/L, methanol, 293 K). From left to right are the colored solution, the photo-bleached solution and the solution after visible light withdrawn for about 1 min. (c) Absorption spectrum of the MIC (50 µmol/L) before and after adding La^{3+} ions (90 mmol/L).The absorption variation of MIC at 527 nm with La^{3+} ions concentration (Inset). (d) The structure of DMNPSP and its UV responsiveness and UV-assisted metal ion chelation. (e) Photos of DMNPSP solution (2.5×10^{-4} mol/L, THF, 293 K) (middle) and its color change after UV irradiation (left) or adding zinc ions (50 mmol/L) (right). (f) Absorption spectrum of DMNPSP solution (50 µmol/L, THF, 293 K) before and after UV irradiation or adding zinc ions (10 mmol/L).

Previous DFT calculations have shown that the introduction of electron donating groups at 6-position of benzopyran part will make the isomerization from SP to MC forms more difficult [83]. However, the SP and MC forms are still in equilibrium in spite that the equilibrium constant might be very small. Once Zn^{2+} ion was added, the Zn^{2+} ion forms coordination complexes (MC-Zn) with MC forms. As a result, the concentration of MC forms will be reduced thus shifting the equilibrium to the right side (Fig. 3a). Therefore, more and more MC-Zn complexes will form when the concentration of Zn^{2+} ions increased. Upon light irradiation, the MC-Zn coordination bonds are ruptured, releasing the free Zn^{2+} ions. The MC forms of SPs are then transformed back to SP forms, accompanied by color changes. The single crystal structure of MIC-Zn complex indicates that carbonyl group of the ester group and the oxygen atom bonded to spirocarbon atom coordinate with zinc chloride (Fig. S30 and Table S3 in Supporting information). This result confirms that with the assistance of metal ions, the new spiropyran changed from SP to MC-form.

Since the MC to SP process is governed by spiropyran-merocyanine equilibria, the binding strength of coordination complexes will have significant influence on the photochromic prop-

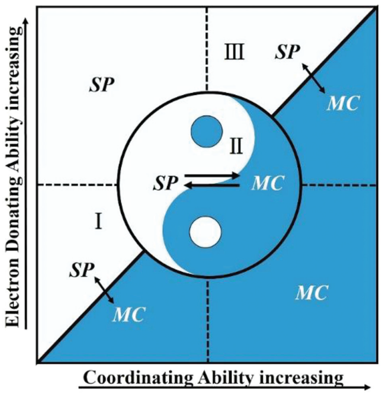

Fig. 4. The influence of different electron donating ability and coordinating ability on spiropyran. Region I: weak electron donating ability and coordinating ability, easily transition between SP and MC, but poor stability; Region II (Tai Chi diagram): moderate electron donating ability and coordinating ability, readily accessible transition between SP and MC and bistable properties; Region III: strong electron donating ability and coordinating ability, very stable in SP or MC state, but the transition requires high energy.

erty. We substituted the Zn^{2+} with La^{3+} as La^{3+}has stronger coordination ability with oxygen atoms and can therefore increase the coordination tendency [84–88]. As shown in Fig. 3c and Fig. S31 (Supporting information), after adding La^{3+} ions, a more significant absorption peaks and color change were observed (Figs. 3b and c, Table S2), indicating that more SP forms were converted to MC forms. However, the La^{3+}-merocyanine complex is too stable so that the colorless solution formed upon irradiation with visible light quickly turned back to red once the irradiation stopped (Figs. 3b and c, Video S4 in Supporting information). We also studied the influence of coordinating ligands, by synthesizing a reported pyridine-containing spiropyran [34] which might form a tris-chelating site, namely 3′,3′-dimethyl-6-nitro-1′-(pyridin-2-ylmethyl)spiro[chromene-2,2′-indoline] (marked as DMNPSP, Fig. 3d and Scheme S5 in Supporting information). After adding Zn^{2+} ions, no color change of the solution was observed, indicating that metal coordination cannot induce SP to MC switching (Figs. 3e and f). This is due to that the pyridine and phenolate anion are too far away from each other and cannot stabilize the coordination complex through chelating effect alone. The switching processes has to rely on UV light (Figs. 3e and f, Fig. S32 in Supporting information), thus may inducing severe photo-degradation. These results indicate that coordination bonds with moderate strength are crucial to achieve reversible spiropyrans without UV light used.

Based on the results mentioned above, a tentative relationship between the structure and property of such spiropyrans can be derived. According to the 6-position substituent effects (such as electron donating ability) on the spiropyran ring-opening reaction reported before [89–92] and the aforementioned influence of coordinating ability, the spiropyrans can be classified into several types (Fig. 4). If the spiropyran compounds possess strong electron donating ability and weak coordinating ability (upper left corner area), or the opposite (lower right corner area), they will exhibit single conformation and are hard to undergo photo-isomerization. Even if they undergo photoswitching process under high energy irradiation, they will change to initial state quickly. In region I with both weak electron donating capability and coordinating capability, the transition between SP and MC is very easy, but both states have low stability. In region III, both electron donating ability and coordinating ability are strong, the compounds will be very stable in both the SP and MC states, but the transition between two states would be very difficult and need high energy irradiation. An ideal photochromic spiropyran compound should better reside in tai chi graphics region II. With moderate electron donating ability and coordinating ability, the transition between SP and MC is readily accessible due to the lower activation energy, but both the SP and MC structure have relatively good stability, thus leading to bi-stability or fatigue resistance. Such guidelines, if further validated by more experimental data, can be used for further design of spiropyran compounds with improved performances.

In summary, we have successfully synthesized spiropyrans MIC and OIC with electron donating groups and coordinative methyl ester. These compounds do not response to UV light and show remarkable color change after the addition of metal ions. However, the metal complexes of the novel spiropyrans exhibit negative photochromic properties and are responsive to visible light or even daily sunlight. The metal ions complex of two spiropyrans are relatively stable in dark with long bleaching time and coloring time. In addition, compared with conventional spiropyrans, the fatigue resistance of the new spiropyrans are improved. A tentative guideline for future designing stimuli-responsive and tunable bistable spiropyran compounds is provided based on the result of this work. We envisage this strategy based on weak coordinated bonds could lead to more research about stable chromatic materials.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

This work was supported by the National Natural Science Foundation of China (Nos. 21631006 and 21771100).

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.04.055.

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