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Review

Regulation Strategies of Dynamic Organic Room-Temperature Phosphorescence Materials

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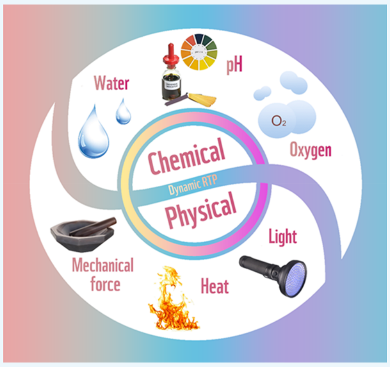
ABSTRACT: Recently, organic room-temperature phosphorescence (RTP) materials, especially those with reversible responses to external stimuli, have attracted extensive attention. A dynamic regulation strategy enables the materials to rapidly respond to external stimuli, gifting varied RTP performance and greater application potential in sensitive sensing, detection, and so on. For these reasons, this Review summarizes progress in the regulation of dynamic RTP in recent years. It focuses on physical regulatory factors including light, heat, and mechanical force as well as chemical regulatory factors including water, pH, and oxygen. It is expected to be beneficial for developing smart materials with dynamic RTP in the future.



KEYWORDS: *room-temperature phosphorescence, dynamic regulation, smart materials, stimulus responsiveness*

■ INTRODUCTION

Organic room-temperature phosphorescence (RTP) has received wide attention from researchers because of its long lifetime and large Stokes shift.[1,2] As shown in Figure 1A, when a molecule absorbs light in the ground state, its electrons transition from the ground state to different energy levels of the excited singlet state and rapidly release part of the energy through non-radiative deactivation, reaching the lowest excited state.[3−8] If the excitons undergo spin inversion, then the lower vibrational energy level of the singlet state will overlap with the higher energy level of the excited triplet state. It is possible that intersystem crossing (ISC) will occur and excitons will reach the excited triplet state. Next, the excitons will go back to the ground state to emit phosphorescence by radiative transitions. However, compared with fluorescence generated from radiative transitions of the singlet state, the triplet excited state that produces phosphorescence is more susceptible to deactivation by environmental influences such as oxygen, which increases the difficulty of obtaining phosphorescence.

Most molecules, due to weak spin−orbit coupling (SOC) and a small ISC rate, are not conducive to emitting phosphorescence (Figure 1B). In consideration of the phosphorescence mechanism, two approaches have been proposed to construct an effective RTP system:[9−12] (1) promoting singlet−triplet ISC to generate more triplet excitons by introducing halogen atoms and heteroatoms and (2) suppressing the non-radiative attenuation via constructing a rigid micro-environment. For the first approach, heavy atoms (Cl, Br, I) and carbonyl or heteroatoms (N, S, P) can enhance SOC and further boost ISC. Unfortunately, the strong SOC produces a high phosphorescence quantum yield (QY) by increasing the ISC rate but decreases the phosphorescence lifetime by promoting radiative transition. Therefore, how to balance the QY and lifetime of phosphorescence is becoming an increasingly important problem. As is well known, the triplet state is easily inactivated by molecular collisions or quenching factors (oxygen) in the environment during the slow radiation process of phosphorescence. So, approaches were proposed to alleviate the non-radiative inactivation of triplet excitons, such as crystal engineering,[13−17] host−guest doping,[18−21] H-aggregation,[22−25] polymerization,[26−29] and supramolecular interaction,[30−35] all of which can construct a rigid environment to effectively limit the molecular vibration and isolate quenching factors. Based on these two methods, many RTP systems with high QY and long lifetime have been constructed.[36−39]

With the development of RTP materials, traditional RTP materials are not the only pursuits. Smart materials with a reversible RTP are becoming a new hot topic. For stimulus-responsive RTP materials, luminescence parameters, including wavelength, lifetime, and intensity, can be adjusted by external

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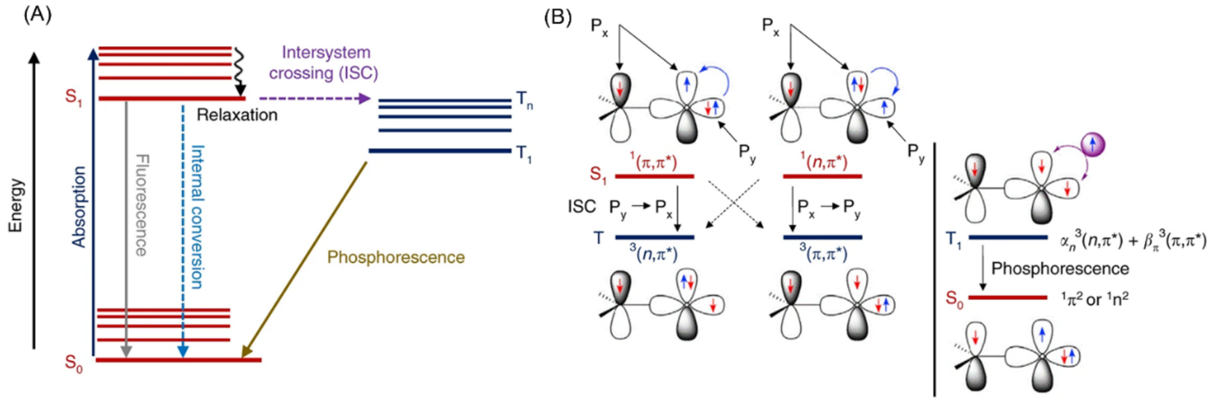

Figure 1. (A) Simplified Jablonski diagram: fluorescence is an emission from an excited singlet state (S_1) and phosphorescence from the triplet state (T_1) after an intersystem crossing (ISC) from the S_1 state. Reproduced with permission from ref 5. Copyright 2019, Springer Nature. (B) Illustration of El-Sayed's rule, the heavy atom effect, and a hybrid atom containing lone-pair electrons. Reproduced with permission from ref 6. Copyright 2016, Elsevier.

factors. Because of their good reversibility and fast responsiveness, smart RTP materials have better potential for applications in imaging,[40,41] anti-counterfeiting,[42−44] encryption,[45,46] sensing,[47,48] and other fields.[49−51] In recent years, although research on smart RTP materials is becoming a hot topic, no systematic summary has reported. Here, we summarize recent progress in the reversible regulation of RTP and focus on chemical and physical regulation strategies.

■ PHYSICAL REGULATORY FACTORS

Light. Light is the most direct and non-invasive physical regulatory factor. Photochromic materials are the traditional dynamic smart materials based on photoresponsiveness. Due to their diverse response forms, sensitive responsiveness, simple conditions, and other advantages, light-responsive RTP materials are the most widely studied stimulus-responsive materials. Common photochromic strategies include cycle-opening/closing,[52,53] photo-polymerization/depolymerization,[54] free radical generation/annihilation,[55,56] and so on.[57,58]

Ma et al.[52] proposed a general strategy of controlling the isomerization of the energy receptors to remotely regulate luminescence with different colors. In their work, diarylethene, with strong emission of closed-loop isomers and the non-fluorescent emission of open-loop isomers, was chosen as a perfect candidate for a tunable energy receptor (Figure 2A). Based on the recent discovery of the excellent host−guest RTP system 1BBI−DMBA,[59] the color can be changed by the energy transfer between the acceptor and donor. Radiative energy transfer plays an important role in this process. The difference from the traditional non-radiation energy-transfer process is that the "apparent lifetime" of the energy acceptor is the same as that of the energy donor. Similarly, in 2022, Tian et al.[53] also reported a reversible fluorescence−RTP switch based on dithienylbenzothiophene (Figure 2B). These studies have led to the development of organic phototunable RTP materials and further exploration of the mechanism of reversible phosphorescence.

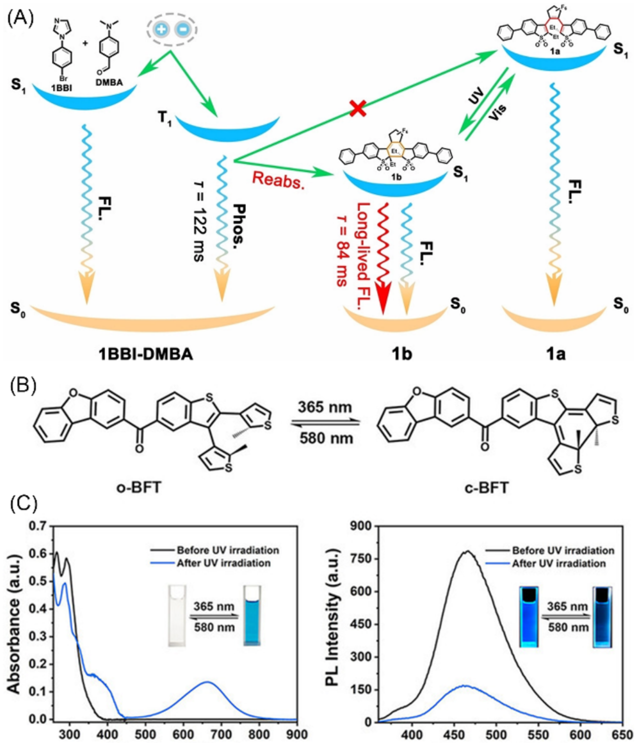

Figure 2. (A) Diagram for the mechanism of the tunable afterglow based on radiative energy transfer. Reproduced with permission from ref 52. Copyright 2021, John Wiley and Sons. (B) Reversible structural isomerization between the opened conformer and the closed photo-isomer. (C) Absorption and PL emission spectra of o-BFT and c-BFT. Reproduced with permission from ref 53. Copyright 2021, John Wiley and Sons.

In recent years, dynamic covalent chemistry has attracted wide attention. Based on supramolecule-mediated [4+4] photo-cyclodimerization of anthracene, Ma et al.[54] successfully designed and prepared two novel copolymers, Poly-AC-CD and Poly-An-CD, which realized the transformation of reversible dynamic covalent bonds (Figure 3A). The polymers exhibit good RTP emission and achieve color conversion between blue and cyan, providing a new method for the design and synthesis of organic dynamic RTP materials.

In 2021, Ma et al.[55] designed two polymers containing HABI, which is a kind of photochromic structure[60] with dual-mode emission of fluorescence and phosphorescence. In addition, both polymers undergo sensitive photochromic reactions under UV light. The polymers have good reversibility, and their color can transform between faint yellow and brown under UV excitation and heating (Figure 3B). This work provides a potential way to obtain RTP

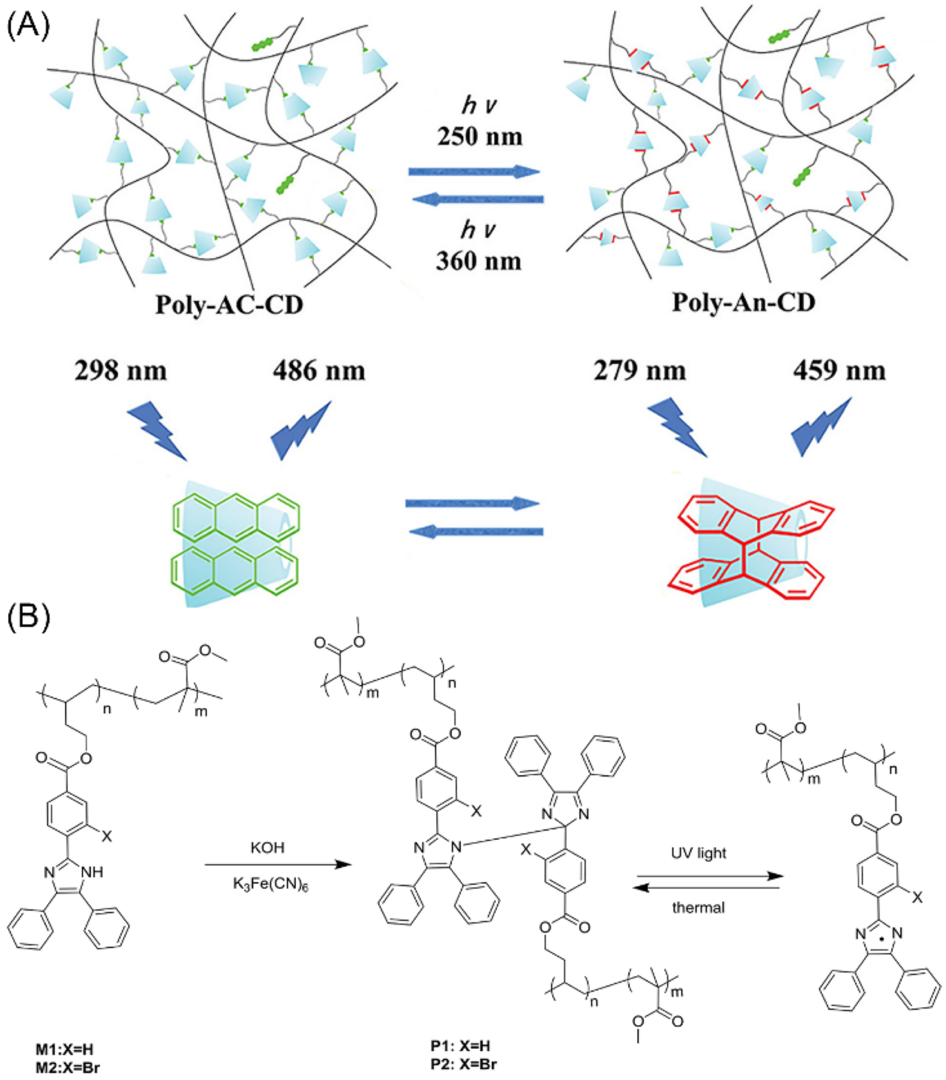

Figure 3. (A) Dynamic photopolymerization system based on anthracene, supramolecule-mediated. Reproduced with permission from ref 54. Copyright 2021, John Wiley and Sons. (B) Photocontrollable room-temperature phosphorescence of organic photochromic polymers based on hexaarylbiimidazole. Reproduced with permission from ref 55. Copyright 2021, Science China Press and Springer-Verlag GmbH Germany, part of Springer Nature.

materials by introducing photochromic molecules into rigid polymer substrates. Similarly, Ding et al.[56] obtained a photoreversible RTP polymer by copolymerizing benzothiadiazole derivatives with acrylamide.

It is well known that[61] the introduction of substituent groups can change the arrangement and stacking of molecules. Chen et al.[62] constructed pure organic small molecules with a stimulus-responsive circularly polarized ultralong RTP (CP-OURTP) by introducing chiral ester chains into phosphorescent carbazole units (Figure 4A). The flexible chains can be used not only as chiral units to realize circularly polarized luminescence emission but also as conformational regulatory factors to effectively change molecular arrangements and stacking patterns. They found that an external stimulus could drive flexible chains to dynamically adjust molecular stacking to emit differently. The stimulus-response behavior is reversible, resulting in the dynamic transformation of photoactivated CP-OURTP and CP-RTP.

Heat. Due to the high sensitivity of the triplet state to heat, a high temperature is not conducive to phosphorescence emission. Thus, temperature has been used as a regulation strategy to achieve reversible phosphorescence.[63] The thermal reversible reaction is an advanced strategy for achieving reversible phosphorescence.[64]

In 2020, Wang et al.[63] proposed a novel strategy for RTP emission with high phosphorescence QY in polymer systems. Due to the rigid environment in the polymer, the lifetime of RTP reached 2.28 s and the QY of RTP reached 8.35%. With heating, the RTP weakened and then recovered when the temperature returned to room temperature. The Diels−Alder (D-A) reaction is a [4+2] cycloaddition reaction between dienes and dienophiles under heating. Based on the D-A reaction, Tian and Ma et al.[64] synthesized three polymers that can be reversibly transformed through thermoreversible dynamic covalent bonds (Figure 4B). All polymers show decent RTP emission with different colors, and the highest absolute phosphorescence QY reaches up to 12 %. They suggested that it was possible to achieve a dynamic RTP by regulating the HOMO−LUMO energy gap through reversible D-A reactions. In addition, many other heat-responsive RTP

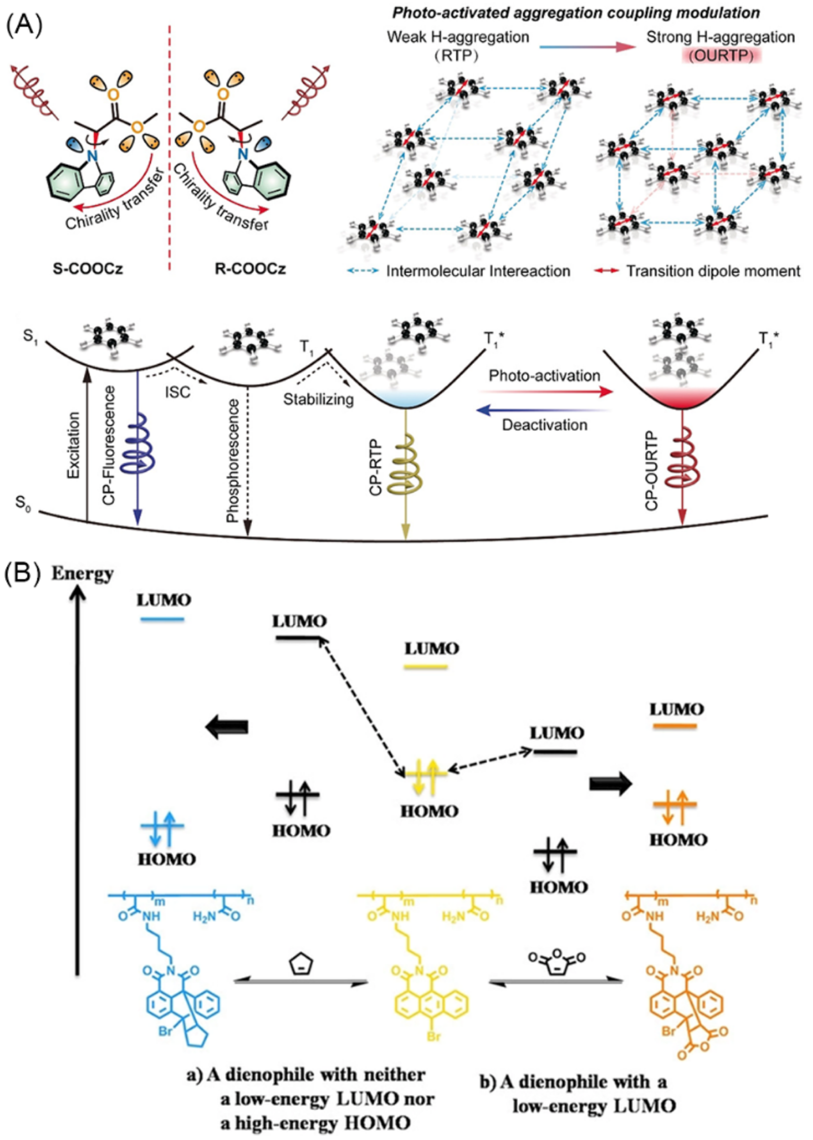

Figure 4. (A) Stimuli-responsive circularly polarized organic ultralong room-temperature phosphorescence. Reproduced with permission from ref 62. Copyright 2020, John Wiley and Sons. (B) Dynamic RTP system based on Diels−Alder reaction. Reproduced with permission from ref 64. Copyright 2020, John Wiley and Sons.

materials, including those based on organic crystals,[16,17] organometallic crystals,[65,66] and liquid crystals,[67−69] have been reported in recent years.

Mechanical Forces. Mechanical force is one of the most convenient physical regulatory factors, as it can be generated without any special requirements. It influences molecular isomerization, packing mode, and conformation, thereby further affecting the luminescence performance.[25] It is reported that triplet excitons are easily affected by quenching factors or molecular motion, resulting in the disappearance of phosphorescence. When a crystal with RTP properties is transformed into an amorphous state by mechanical stimulation such as grinding, the phosphorescence usually weakens with the destruction of the regular molecular arrangement inside the crystal. The destruction of crystal structures is usually recoverable via steam fuming.

Halogen bonding plays an important role in the research on dynamic RTP, being one kind of weak interaction that can be dynamically regulated by external stimuli such as mechanical forces, X-rays, and heat.[70] In addition to this, halogen atoms (bromine and iodine) can also enhance RTP emission. On one hand, halogen bonding enhances SOC and promotes ISC through the heavy atom effect, thus increasing phosphorescence QY. On the other hand, it provides a rigid microenvironment and suppresses non-radiative inactivation. Based on intermolecular halogen bonding of the bromo−cyano groups in organic host−guest systems, Dong et al.[71] proposed a common strategy for the construction of efficient persistent RTP materials with a multi-stimulus responsiveness (Figure 5A,B). Using CBZCN and TPACN as the guest and TPABr as the host, respectively, they obtained the dual-mode emission of thermally activated delayed fluorescence (TADF) and RTP. The intensity ratio of fluorescence and phosphorescence varied with the doping ratio and excitation wavelength. Thus, they achieved a large-scale multicolor emission from blue to red, including white (CIE: 0.33,0.32).

In 2020, Li and Tang et al.[72] developed a novel strategy for constructing dynamic RTP with stimulus responsiveness based

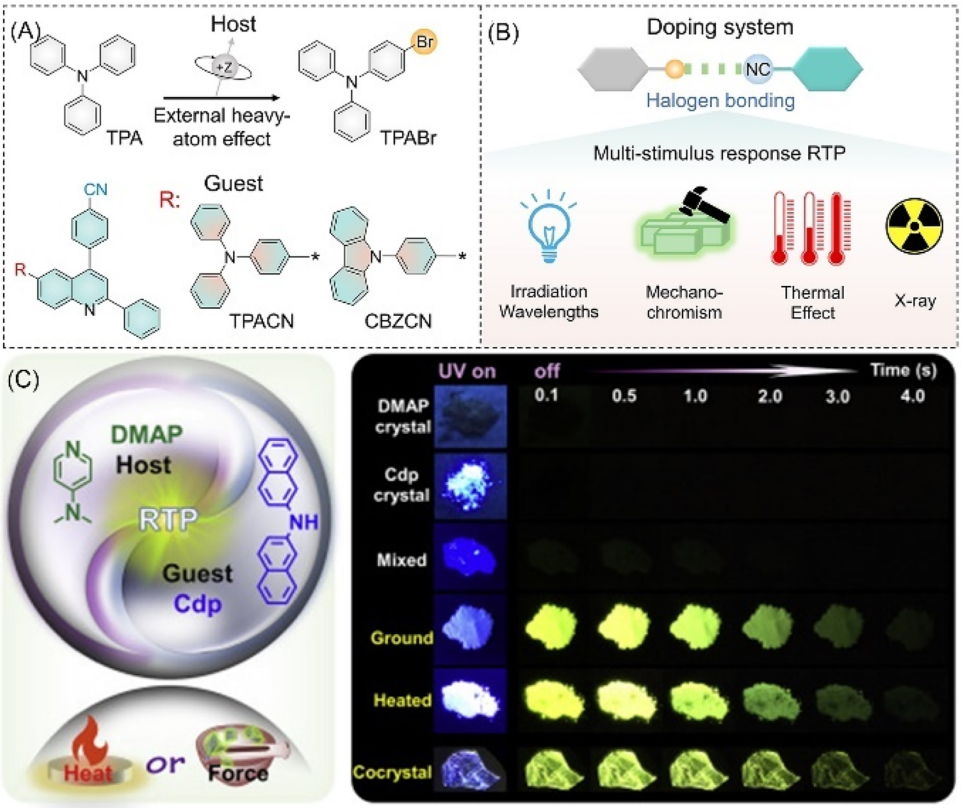

Figure 5. (A) Chemical structure of the organic host matrices and guest molecules. Reproduced with permission from ref 71. Copyright 2022, John Wiley and Sons. (B) Schematic diagram of stimulus-responsive RTP in the host−guest doping system through weak intermolecular halogen bonding. Reproduced with permission from ref 71. Copyright 2022, John Wiley and Sons. (C) Stimulus-responsive RTP system based on FRET. Reproduced with permission from ref 72. Copyright 2020, Elsevier.

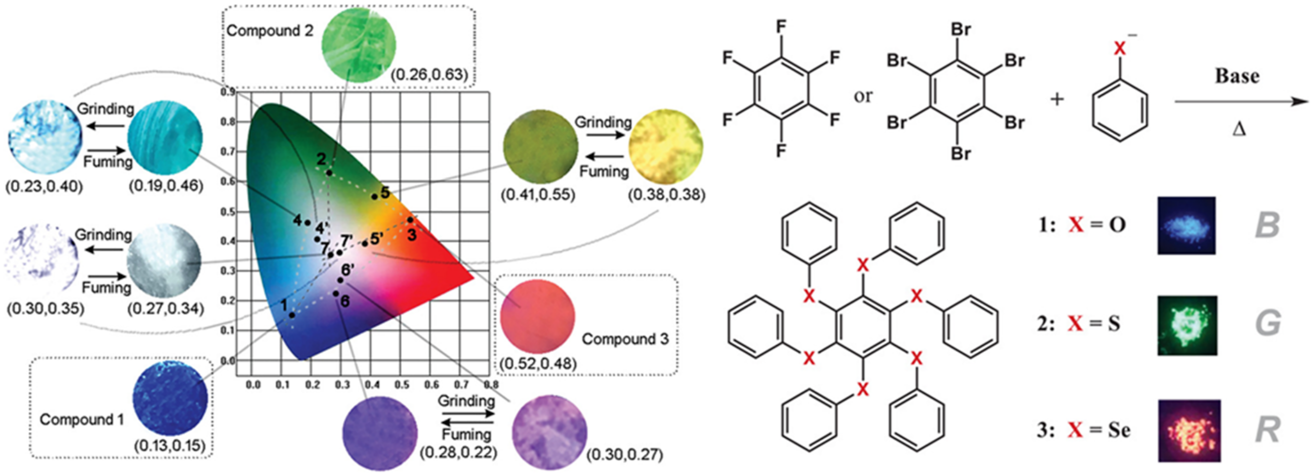

Figure 6. Illustration of the chemical structures and CIE 1931 chromaticity diagram (Dots 1 and 3 signify the luminescent color coordinates for the corresponding film states of compounds **1**−**3**, respectively. The dots 4−6 feature the luminescent color coordinates for the film states of mixtures (**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. Reproduced with permission from ref 78. Copyright 2020, John Wiley and Sons.

on Förster resonance energy transfer (FRET) (Figure 5C). As we know, the efficiency of FRET increases as the intermolecular distance decreases.[73] Mechanical forces are usually used to change the intermolecular distance, thus affecting the FRET process. In general, grinding will narrow the distance between the host and the guest.[74] They selected 4-dimethylaminopyridine (DMAP) and 2,2-dinaphthylamine (Cdp) as the host and guest, respectively, to realize a reversible yellow RTP with a lifetime up to 935 ms.

It is reported that dynamic luminescence with a substantial color difference can be achieved through the dual emission of fluorescence and phosphorescence.[75] In 2017, Zhu et al.[76,77] developed a series of starlike persulfurated arenes with diverse self-assembly behaviors, achieving precise regulation of

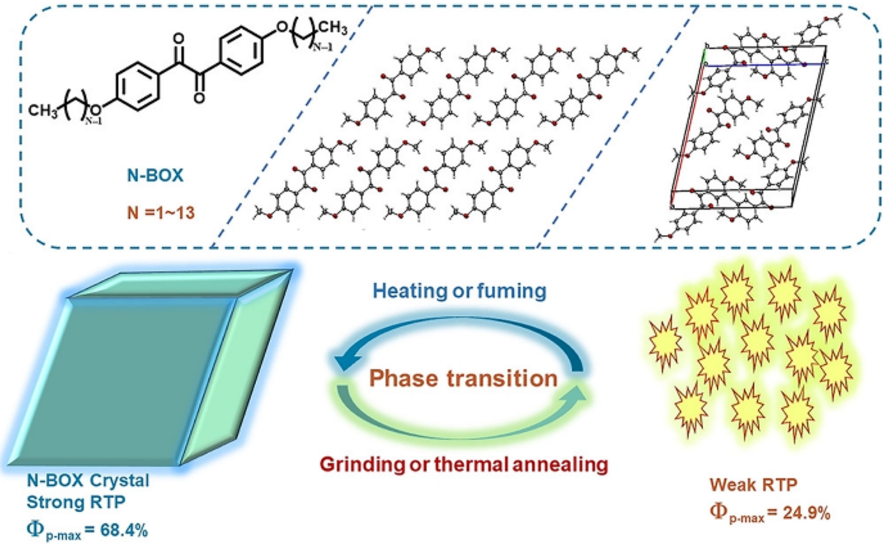

Figure 7. (Top) Molecular structure of N-BOX (left) and unit cell of 1-BOX in a crystal (middle and right), and (bottom) schematic diagram of N-BOX stimuli-responsiveness of RTP as a result of phase transition (bottom). Reproduced with permission from ref 79. Copyright 2022, John Wiley and Sons.

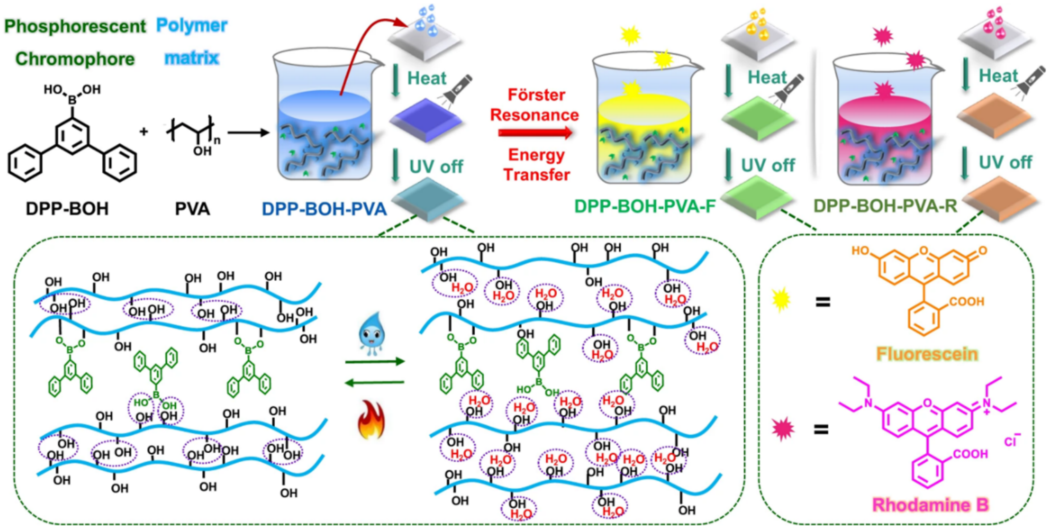

Figure 8. Schematic illustration of changes in intermolecular interactions under heating or water stimulus.

fluorescence and phosphorescence. Later they[78] added the O, S, and Se atoms into the starlike structure. In Figure 6, the luminescent colors of the membrane states corresponding to compounds **1**−**3** are respectively blue, green, and red. By mixing these luminescent membranes, it is possible to achieve panchromatic emission. The luminescence in this system can be reversibly changed by grinding and fuming. In addition, the tunable dual-mode emission of fluorescence and phosphorescence can also be obtained by a physical cocrystal strategy.

Recently, Tian and Ma et al.[79] successfully developed a series of 1,2-bis(4-alkoxyphenyl)ethane-1,2-dione derivatives (N-BOX), shown in Figure 7, exhibiting multi-responsiveness. Among these derivatives, the compound 7-BOX demonstrates a high RTP QY, reaching an impressive 68.4%. Furthermore, apart from their remarkable mechanical responsiveness, N-BOX derivatives also exhibited a notable thermal sensitivity. The strong blue RTP of the crystal can be transformed to the weak yellow RTP of amorphous material through grinding. The RTP emission of N-BOX in the crystalline state was easy to adjust by external stimuli (grinding or thermal annealing), which led to a phase transition and generated unique multilevel stimuli-responsiveness. The length of the alkyl chain had a great influence on the RTP emission. Experimental results demonstrated that longer chain lengths led to larger blue shifts, consistent with a previous report.[80] Building upon these findings, Ma and colleagues[81] further offered a novel strategy for designing and constructing elastic crystals with stimulus-responsive RTP capabilities.

■ CHEMICAL REGULATORY FACTORS

Compared with physical regulatory factors, chemical regulatory factors encompass a broad scope of chemical substances and involve intricate chemical reactions. Therefore, this Review provides a concise overview of the recent advances in dynamic RTP pertaining to various common chemical regulatory factors such as water, oxygen, acids, and bases.

Water. It has been reported that quenching effects and non-radiative deactivation play important roles in constructing

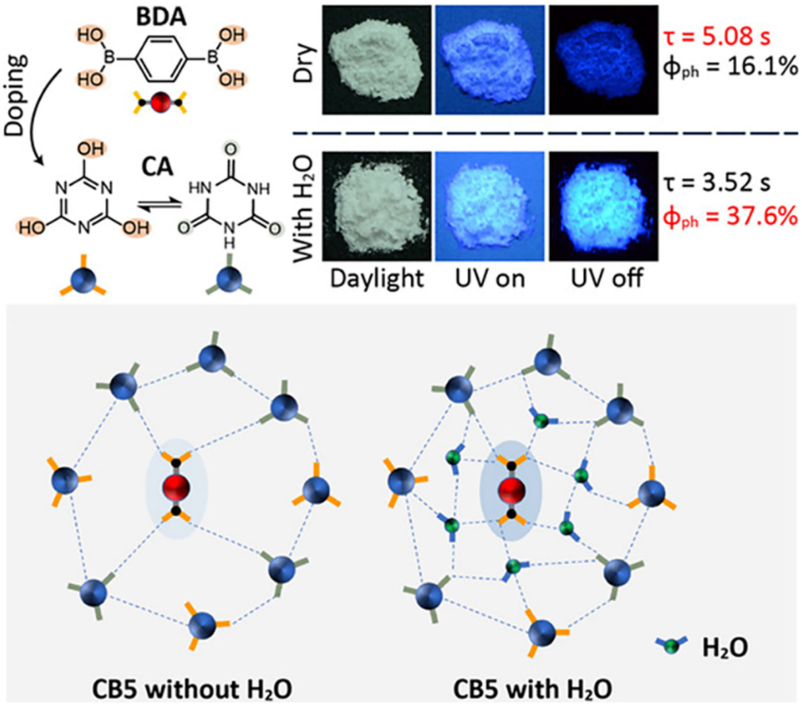

Figure 9. Water-responsive deep-blue organic ultralong phosphorescence system based on DBA. Reproduced with permission from ref 88. Copyright 2021, John Wiley and Sons.

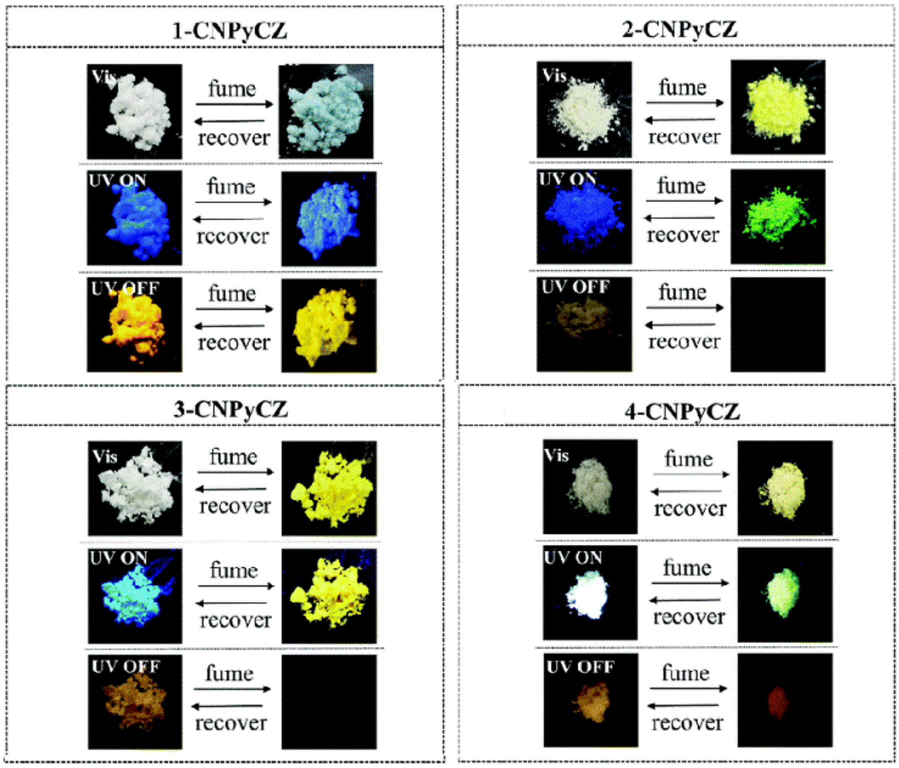

Figure 10. Color changes of the initial crystalline powders upon fuming and recovering. Reproduced with permission from ref 89. Copyright 2021, The Royal Society of Chemistry.

dynamic water-responsive RTP materials.[82] Thus, the dynamic water-responsive RTP materials exhibit immense potential for the sensing and detection of water owing to their heightened sensitivity and responsiveness.

Because the hydrogen bond network of the polymer can be easily destroyed by water, a conventional approach is to fabricate a water-sensitive RTP system by polymerization. Recently, Li and Tang et al.[83] developed water-responsive RTP materials by utilizing the B−O covalent bond between arylboronic acid phosphor and poly(vinyl alcohol) (PVA) matrix; the structures are shown in Figure 8. The B−O covalent bond is susceptible to water. Consequently, the

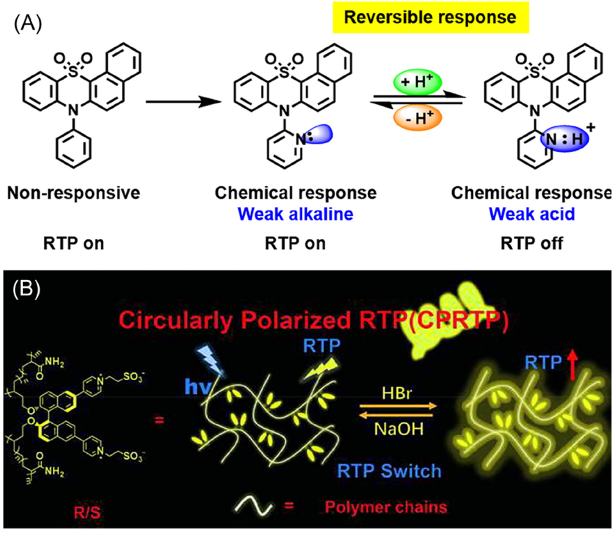

Figure 11. (A) Acid−base reversible RTP system based on the pyridine structure. Reproduced with permission from ref 90. Copyright 2021, John Wiley and Sons. (B) Illustration of acid/base-responsive structure of *R*/*S*-polymers. Reproduced with permission from ref 91. Copyright 2021, Elsevier.

researchers achieved a reversible water-responsive RTP material by employing water−steam fuming and heating. In addition, fluorescein and rhodamine B have been incorporated to achieve multicolor luminescence via triplet-to-singlet Förster resonance energy transfer (TS-FRET). Although the presence of a rigid covalent bond within the polymer system restricts the molecular motion of the phosphors and enhances RTP,[84] it concurrently diminishes their sensitivity toward stimulus.

Polyacrylamide (PAM), being the most prevalent and crucial polymer system, has gained widespread recognition for its remarkable performance. In 2021, Ma et al.[85] introduced a facile secondary processing strategy that utilized water molecules as hydrogen bridges to significantly improve RTP emission. They developed an imidazolium−naphthalene derivative and copolymerized it with acrylamide to produce a dynamic RTP copolymer. This copolymer effectively utilizes water molecules in its surrounding environment as hydrogen bridges to enhance rigidity, thereby improving the efficiency of RTP.

In addition to polymerization, organic small-molecule doping can also achieve specific water-responsive behavior. Notably, boron exhibits promising potential in achieving ultralong RTP without the heavy atoms due to its empty p-orbital and electron-accepting characteristics.[86,87] In 2021, Chen et al.[88] demonstrated an ultralong deep blue RTP achieved by simply doping 1,4-benzenediboronic acid (BDA) into cyanuric acid (CA) (Figure 9). This material displayed excellent stability at room temperature and possessed an impressive RTP lifetime of up to 5.08 s. Interestingly, the QY significantly increased from 16.1% to 37.6% as the water content rose from 0% to 20%. According to their perspectives, there are two forms of CA: enol and keto. The keto form is more stable and predominant in the doping system. Serving as an exceptional candidate, the material forms a compact and rigid hydrogen bond network upon addition of water, promoting efficient RTP emission by suppressing non-radiative transitions.

Acid or Base. In an acid/base-responsive RTP system, the conventional approach utilizes protonation and deprotonation of nitrogen atoms. In this way, Liu et al.[89] synthesized four types of acid-responsive D-A-A′ molecules by employing the pyridine ring as an acceptor and carbazole as a donor (Figure 10). Li et al.[90] developed a novel host−guest doping system using triphenylphosphine oxide ($OPPh_3$) as the host and benzo(dibenzo)benzothiazine derivatives as the guest which exhibits stimulus-responsive RTP properties. By introducing the pyridine structure into the guest molecule, dynamic RTP can be achieved (Figure 11A).

The sulfo group plays a crucial role in these acid-responsive RTP systems. The sulfur−oxygen double bond of SO^{3-} not only enhances the SOC but also forms extensive hydrogen bonds with the polymer matrix, effectively suppressing non-radiative transitions. Taking inspiration from this, Ma et al.[91] obtained a pure organic amorphous RTP polymer, *R*/*S*-BPNaP, with circularly polarized luminescence through radical copolymerization of acrylamide and chiral dinaphthyl derivatives (Figure 11B).

The realization of near-infrared (NIR) organic RTP has posed a significant challenge, garnering considerable attention from researchers due to its immense potential in biological imaging and other domains.[92] It is a pity that reports of the reversible regulation of NIR-RTP are still limited. It has been reported in the literature[93] that the more electron delocalization in a molecule with D-A structure, the more favorable it is to producing emission with longer wavelengths. In 2022, Ma et al.[94] reported a PVA film doped with phenolsulfonephthalein

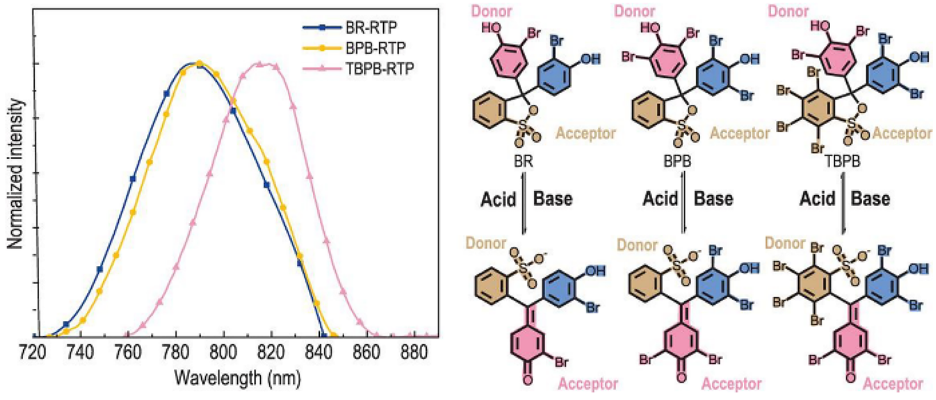

Figure 12. RTP emission of PSP@PVA-N (λ_{ex} = 550 nm) and structural transformation of BR, BPB, and TBPB in aqueous solution with differing pH values and their charge distribution in the ground state (S_0).

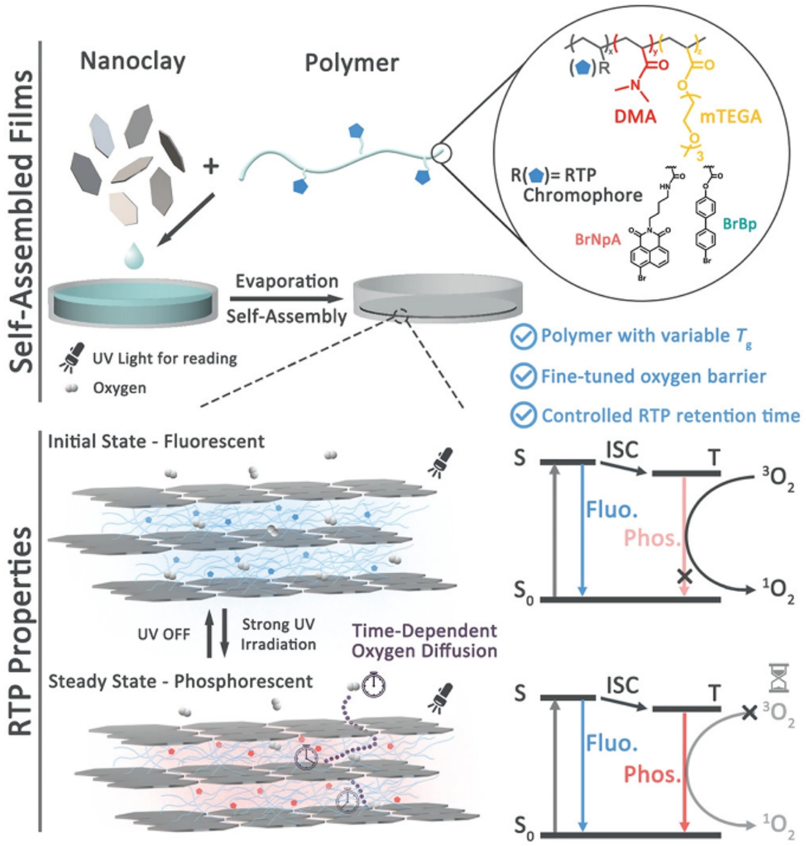

Figure 13. Schematic illustration of the preparation and structure of the RTP-copolymer/nanoclay nacre-mimetic nanocomposite. Reproduced with permission from ref 95. Copyright 2020, John Wiley and Sons.

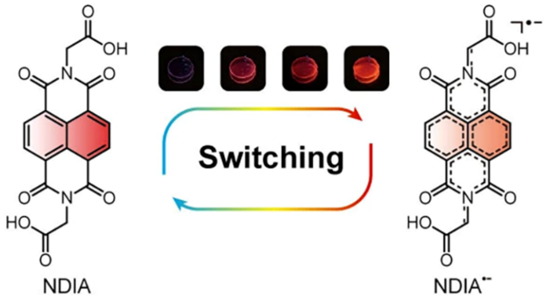

Figure 14. Dynamic RTP behavior strategy through free radical formation. Reproduced with permission from ref 96. Copyright 2022, Science China Press and Springer-Verlag GmbH Germany, part of Springer Nature.

(PSP) derivatives, which have reversible acid/base-responsive NIR-RTP emission. As depicted in Figure 12, the PSP derivatives exhibit an annular structure when exposed to an acidic environment and undergo deprotonation to open the ring under neutral and basic conditions, establishing a prerequisite for constructing acid/base-responsive materials. The open-loop state (PSP@PVAN) and closed-loop state (PSP@PVA-H) of PSP derivatives can be controlled in situ by introducing acids and bases into the PVA matrix, showing high pH sensitivity of RTP emission.

Oxygen. It is known that the presence of oxygen in the environment quenches phosphorescence. Thus, it is crucial to effectively isolate oxygen to construct an RTP system. Recently, Tian et al.[95] proposed an innovative approach to incorporate RTP polymer into nanoclay composite materials, developing a cost-effective and practical film with excellent RTP properties (Figure 13). The lamellar structure of the nanoclay acts as an oxygen barrier, effectively mitigating the quenching effect caused by ambient oxygen. It is possible to regulate the oxygen content within the film by precisely adjusting the ratio of nanoclay and polymer, enabling its application in information storage and anti-counterfeiting measures. In 2022, An et al.[96] reported a novel dynamic RTP through free radical generation/annihilation (Figure 14). When NDIA was doped into the PVA matrix, it showed strong RTP emission. With an increase in the irradiation time, NDIA molecules gradually underwent conversion into NDIA anion radicals, accompanying the decrease in RTP emission intensity. Further, the RTP can be recovered after the oxidation of materials and has good repeatability.

■ SUMMARY AND OUTLOOK

Here we present a brief overview of recent advances in the regulatory factors of dynamic RTP, with a specific focus on physical factors, including light, heat, and mechanical force, as well as chemical factors encompassing water, pH, and oxygen. Smart dynamic RTP materials will have a more important role in practical applications in the future. In our perspective, exploring the underlying mechanism of dynamic RTP emission will greatly facilitate the progress toward smart luminescent materials. This Review outlines diverse regulatory factors and mechanisms commonly employed in recent years, aiming to offer valuable insights for advancements in smart RTP materials in the future.

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Notes

The authors declare no competing financial interest.

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