

ARTICLES

Polyrotaxane-derived unique supramolecular mechanoresponsive elastomers

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Abstract Two bridged core-brominated pyromellitic diimide (BrPMDI) derivatives functionalized with four or two ethylene glycol side chains (BrPMDI-1-B and BrPMDI-2-B) are synthesized and copolymerized with 2-(2-methoxyethoxy)ethyl methacrylate (MEP) and low-coverage polyrotaxane (Pr) to construct supramolecular elastomers with distinct mechano-enhanced or mechanochromic pure phosphorescence. A facile side-chain shortening strategy enables a switch of BrPMDI from a luminescence-quenching to an emissive molecule; that is, BrPMDI-2-B exhibits orange phosphorescence in the solid state, whereas BrPMDI-1-B suffers from luminescence quenching. Single-crystal structural analysis reveals that increased side-chain length strengthens π - π stacking and disrupts intermolecular halogen bonds, leading to luminescence weakening or even quenching. Significantly, the elastomer 5 wt%-BrPMDI-1-B-0.5 wt%Pr shows a 2.2-fold enhancement in green phosphorescence intensity (500 nm) upon stretching, while 5 wt%-BrPMDI-2-B-0.5 wt%Pr undergoes a phosphorescence color transition from orange (600 nm) to green (500 nm). In addition, the bridged architecture not only promotes chromophore aggregation but also endows the elastomers with intrinsic reversible mechanoresponse. Furthermore, Pr incorporation improves the tensile strain of the elastomers by 1.2-fold, facilitating the thorough dispersion of chromophores into a monomolecular state. This work provides novel insights into the molecular design of BrPMDI derivatives and, more importantly, extends stretch-responsive luminescent materials to the realm of pure phosphorescence.

Keywords mechanochromism, pure phosphorescence, polyrotaxane, elastomer, reversible

1 Introduction

Mechanochromic materials leveraging their sensitive response to exogenous forces are progressively penetrating multiple critical fields, including mechanical damage detection [1,2], medical diagnosis [3,4], movement monitoring [5,6], information encryption [7,8] and so on. Especially, mechanochromic phosphorescent materials, benefiting from their long lifetime and large Stokes shift, can effectively minimize background interference from excitation light and short-lifetime fluorescent substances to achieve higher accuracy and reliability of detection. Unfortunately, achieving room-temperature phosphorescence (RTP) presents significant challenges because triplet excitons are easily quenched by oxygen, water and high temperature [9–11]. Therefore, most current research on mechanochromic phosphorescent materials is based on rigid materials such as crystals and powders [12–20]. As compared with flexible and stretchable materials, these rigid materials can only respond through friction, grinding, extrusion and often suffer from non-spontaneous reversibility (requiring heating, light irradiation, solvent fumigation or other means to return to the initial state), a narrow application scope, and complex processing. On the other hand, the realization of flexible mechanochromic phosphorescent materials needs to overcome the contradiction between strong non-radiative transitions caused by flexible chain motion and phosphorescence quenching, as well as the adaptation between the mechanochromic mechanism and flexible deformation. Recently, several attempts were made to solve this

problem [21–25]. Yang and co-workers [24] doped triphenylene derivatives (TPNP/TPNPO) as phosphorescent chromophores into a polyurethane matrix, where mechanical stretching modulates segmental orientation, enabling favorable chromophore-hard segment alignment and leading to stretch-induced phosphorescence enhancement. Dickey and co-workers [25] embedded rare-earth phosphorescent particles, thermochromic dyes, and liquid metal wires (EGaIn) into polydimethylsiloxane (PDMS) to fabricate a flexible “mechanical-thermal-optical” transduction composite, achieving mechanoresponsive phosphorescence intensity modulation. Although these works advance flexible mechanochromic phosphorescence materials, phosphorescence color switching remains unavailable. In addition, these materials are either metal-based (environmentally problematic) or phosphorescence-fluorescence coexisting systems, where intense fluorescence always masks phosphorescence, precluding timely naked-eye observation of force-induced luminescence changes.

Nevertheless, flexible covalently linked mechanochromic pure phosphorescence materials remain scarcely reported. Such materials require chromophores with intrinsic pure phosphorescence and mechanoresponsive properties, including structural perturbations (e.g., aggregation state [26,27], configuration [28,29], bond cleavage [30,31]) upon tensile force, with concomitant phosphorescence modulation. Because core-brominated pyromellitic diimide (BrPMDI) exhibits exceptional pure phosphorescence [32], arising from high ISC efficiency enabled by strong heavy-atom effects and abundant heteroatomic moieties [33], it is widely used as an ideal

building block. In addition, polyrotaxanes, especially low-coverage polyrotaxanes (Pr), can efficiently dissipate local stress and improve stretchability with good reversibility and afford enhanced macro-cycle sliding mobility, leading to pronounced mechanical property improvements.

Therefore, we synthesized two types of bridged core-brominated pyromellitic diimides (BrPMDI) with modified ethylene glycol side chains and copolymerized them with MEP and Pr to prepare supramolecular pure phosphorescent elastomers with reversible mechano-enhancement or mechanochromism (Figure 1), where the bridging architecture promotes molecular aggregation and endows BrPMDI with mechano-responsiveness. Moreover, Pr incorporation significantly improved elastomer elongation, thereby expanding the mechanoresponse range. Ultimately, 5 wt%-BrPMDI-1-B-0.5 wt%Pr achieved a stretch-induced 2.2-fold phosphorescence intensity enhancement, while 5 wt%-BrPMDI-2-B-0.5 wt%Pr exhibited an orange-to-green phosphorescence color transition.

2 Experimental

The materials, methods, and experimental procedures of this work are shown in the Supporting Information online.

3 Results and discussion

3.1 Synthesis and characterization of bridged BrPMDI and Pr

Two bridged BrPMDI with different ethylene glycol side chain

lengths, BrPMDI-1-B and BrPMDI-2-B (bridged), were synthesized via similar procedures. To verify the function of the bridged structure in mechanoresponsive properties, BrPMDI-1-D and BrPMDI-2-D (divided) were synthesized as reference molecules. Their structures and purity were confirmed by proton nuclear magnetic resonance (^1H NMR), ^{13}C NMR, and high-resolution mass spectrometry (HRMS) (Figures S1–S27, Supporting Information online). Pr was synthesized according to a previous report [34]. Based on the peak area integration of the corresponding peaks in ^1H NMR spectra (Figures S28–S29), it was determined that approximately 6 α -cyclodextrin (α -CD) units were threaded onto the polyethylene glycol (PEG) chain ($M_w \approx 10,000$), and each Pr molecule was modified with approximately 8 double bonds.

3.2 Mechanism for side-chain length on the pure phosphorescence behavior of BrPMDI

To elucidate the effect of ethylene glycol unit length on the photophysical properties of BrPMDI, we characterized the excitation spectra, steady-state emission spectra, delayed emission spectra, and corresponding lifetimes of the four compounds in solid powder form (Figure 2a–e). BrPMDI-1-B, a bridged derivative, exhibited luminescence quenching, whereas BrPMDI-2-B showed a dominant emission peak at 610 nm ($\tau = 0.98$ ms, $\lambda_{\text{ex}} = 400$ nm). In contrast, the reference molecules BrPMDI-1-D and BrPMDI-2-D displayed maximum emission at 580 nm ($\tau = 13.24$ ms, $\lambda_{\text{ex}} = 380$ nm) and 610 nm ($\tau = 17.85$ ms, $\lambda_{\text{ex}} = 400$ nm), respectively. These results indicated that increasing the number of ethylene glycol units in

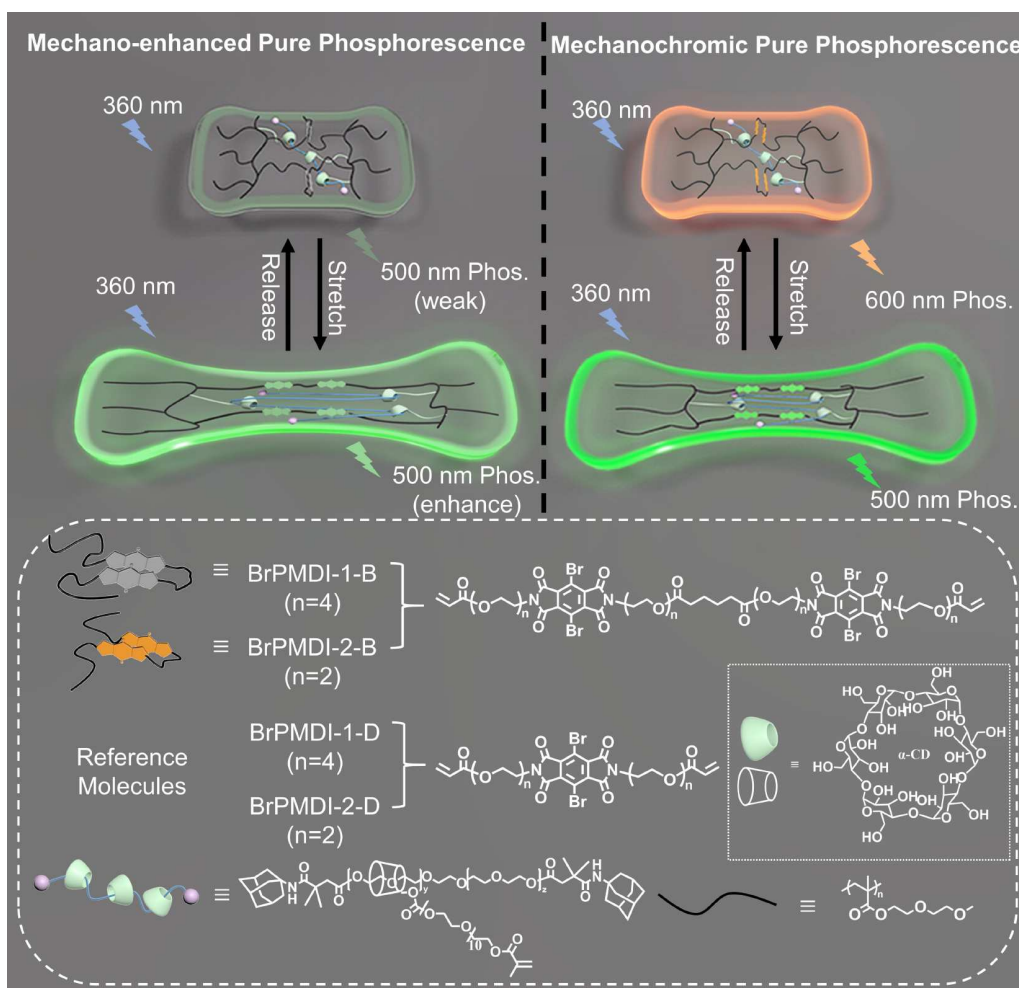


Figure 1 (Color online) Schematic illustration of mechano-enhanced and mechanochromic pure phosphorescence supramolecular elastomers.

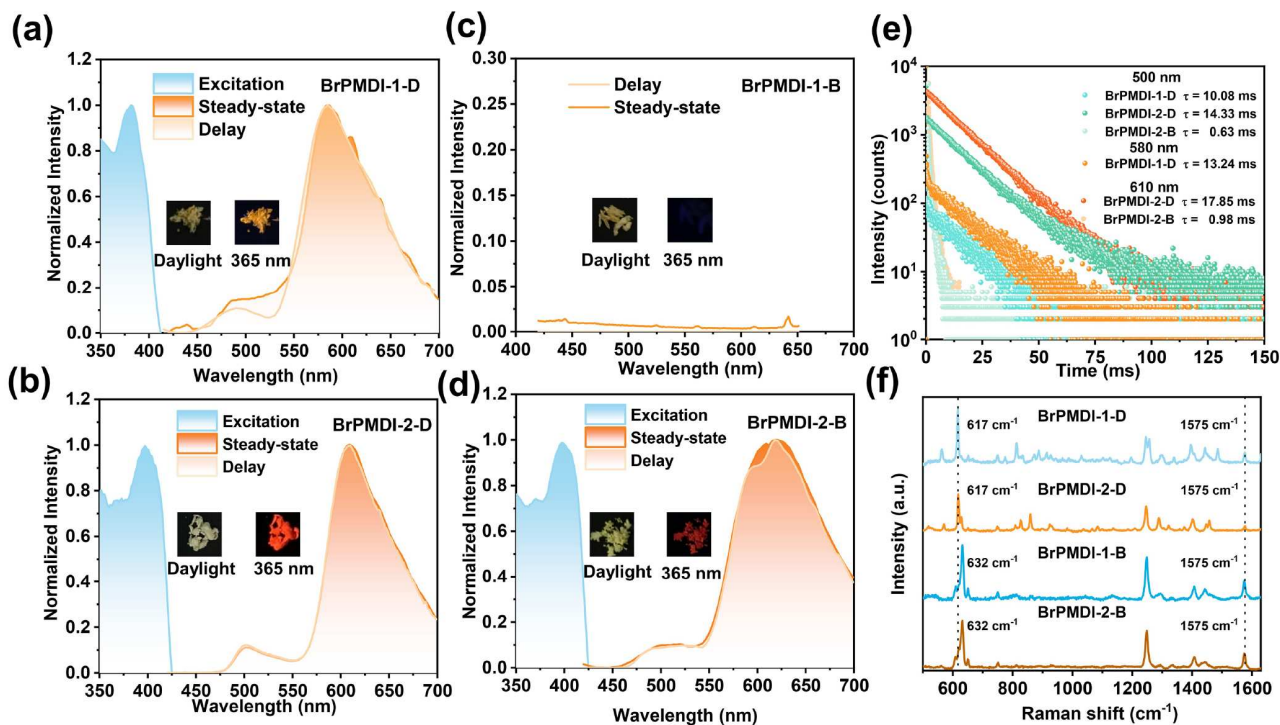


Figure 2 (Color online) Excitation, steady-state emission and delayed spectra (delay time: 0.05 ms) of solid (a) BrPMDI-1-D; (b) BrPMDI-2-D; (c) BrPMDI-1-B; (d) BrPMDI-2-B (inset: photographs of corresponding solid under daylight and 365 nm UV light); (e) time-resolved luminescence decay plots of BrPMDI-1-D, BrPMDI-2-D, and BrPMDI-2-B; (f) Raman spectra of solid.

BrPMDI induced a blue shift of the emission/excitation maxima, accompanied by reduced luminescence intensity and lifetime (Figure S30a). Raman spectra (Figure 2f) revealed C–Br stretching vibrations at 632 cm^{-1} for bridged BrPMDI-1-B and BrPMDI-2-B (with higher intensity for the former) and at 617 cm^{-1} for reference BrPMDI-1-D and BrPMDI-2-D. These observations suggested that increasing ethylene glycol units elevates molecular vibration frequency and intensity, facilitating non-radiative transitions and consequent luminescence attenuation or quenching. To uncover the underlying mechanism, single-crystal X-ray diffraction (SCXRD) was performed. SCXRD structures (Figure 3a, b) showed that adjacent BrPMDI-2-D molecules in the same plane failed to form effective halogen bonds, unlike BrPMDI-1-D, precluding efficient suppression of non-radiative transitions and explaining the reduced luminescence with longer side chains. Additionally, BrPMDI-1-D exhibited a shorter π - π stacking distance ($3.375\text{ \AA}$, $3.420\text{ \AA}$ for BrPMDI-2-D) (Figure 3c, d); and a larger stacking overlap area (30.5% , 11.4% for BrPMDI-2-D) (Figure 3e, f), trending H-aggregation and accounting for the emission blue shift. Notably, the bridged structure intensified molecular flexibility and thermal motion, leading to dynamic and disordered packing, which not only hindered the growth of diffraction-quality crystals but also promoted ineffective packing detrimental to luminescence, resulting in further attenuation or quenching. This hypothesis was validated by the recovery of luminescence from quenched BrPMDI-1-B upon liquid nitrogen freezing (Figure S30b). Subsequently, solution-phase photophysical properties were investigated (Figure S31). All four compounds showed room-temperature luminescence quenching in solution but exhibited a delayed emission peak at 580 nm under low-temperature conditions.

The above results indicated that the side chain length affects the photophysical properties of the aggregated state by altering the molecular packing mode and intermolecular distance, with little influence on the monomolecular state. Meanwhile, the aggregated-state phosphorescence emission of BrPMDI was located at 580 or

610 nm, and the monomolecular state phosphorescence emission was at 500 nm. Importantly, the steady-state spectra and delayed spectra of these emission peaks showed complete overlap, indicating that BrPMDI exhibits rare pure phosphorescent emission behavior both in the aggregated and monomolecular state. This was attributed to the heavy-atom effect of the two bromine atoms on the benzene ring and the lone pairs of electrons introduced by the N and O atoms, which greatly facilitated the intersystem crossing (ISC) efficiency from the singlet excited state to the triplet excited state.

3.3 Aggregation-dependent pure phosphorescence in BrPMDI elastomers

Subsequently, a series of phosphorescent elastomers was prepared by copolymerizing MEP with BrPMDI-1-B or BrPMDI-2-B at varying mass fractions (relative to MEP). Fourier transform infrared (FT-IR) spectra (Figure 4a, b) showed an absorption peak at 1773 cm^{-1} , assigned to the C=O stretching vibration of imide groups, with intensity increasing with chromophore loading. For comparison, analogous elastomers with BrPMDI-1-D or BrPMDI-2-D were prepared under identical conditions. The photophysical properties of these elastomers were further characterized. Excitation spectra of elastomers containing 0.5 wt% of the four chromophores (Figure S32) showed a common maximum at 360 nm (consistent with the monomolecular state), which was used for all subsequent measurements. Delayed emission spectra revealed that as chromophore loading increased from 0.5 to 10 wt%, for BrPMDI-1-B, 500 nm phosphorescence intensity and lifetime gradually decreased (from 55 to 32 μs) (Figure 4c, d), with a concomitant reduction in visible green luminescence (Figure S33a). For BrPMDI-2-B, 500 nm intensity decreased, while a distinct 600 nm peak emerged at 3 wt% and dominated at 5 wt% (Figure 4e), with lifetimes increasing from 0.08 to 3.09 ms (500 nm) and 2.52 to 3.34 ms (600 nm) (Figure 4f), and luminescence color shifting from green to orange (Figure S33b).

These results indicated that increasing chromophore loading drives aggregation from monomers, with emission transitioning from monomer (500 nm) to aggregate (quenching or 610 nm

emission). Elastomers with reference chromophores exhibited similar trends (Figures S33c, d and S34). Despite solid-state orange phosphorescence from aggregated BrPMDI-1-D, no corresponding

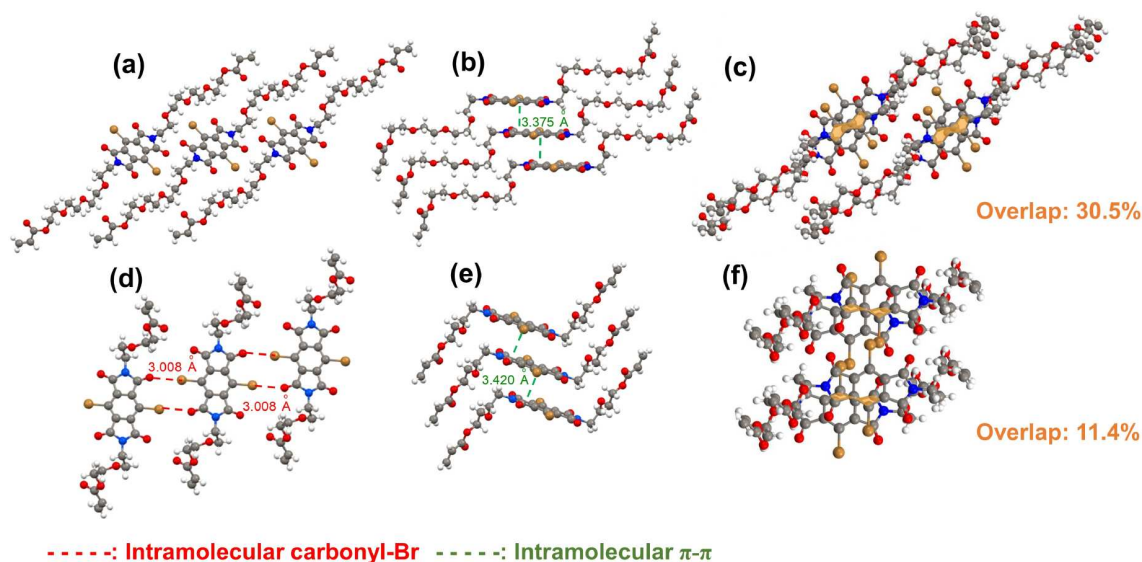


Figure 3 (Color online) Single crystal structures of BrPMDI-1-D (CCDC: 2506170). (a) Intermolecular Br-carbonyl interaction; (b) intermolecular π - π interactions; (c) π - π stacking overlap area. BrPMDI-2-D (CCDC: 2506173). (d) Intermolecular Br-carbonyl interaction; (e) intermolecular π - π interactions; (f) π - π stacking overlap area.

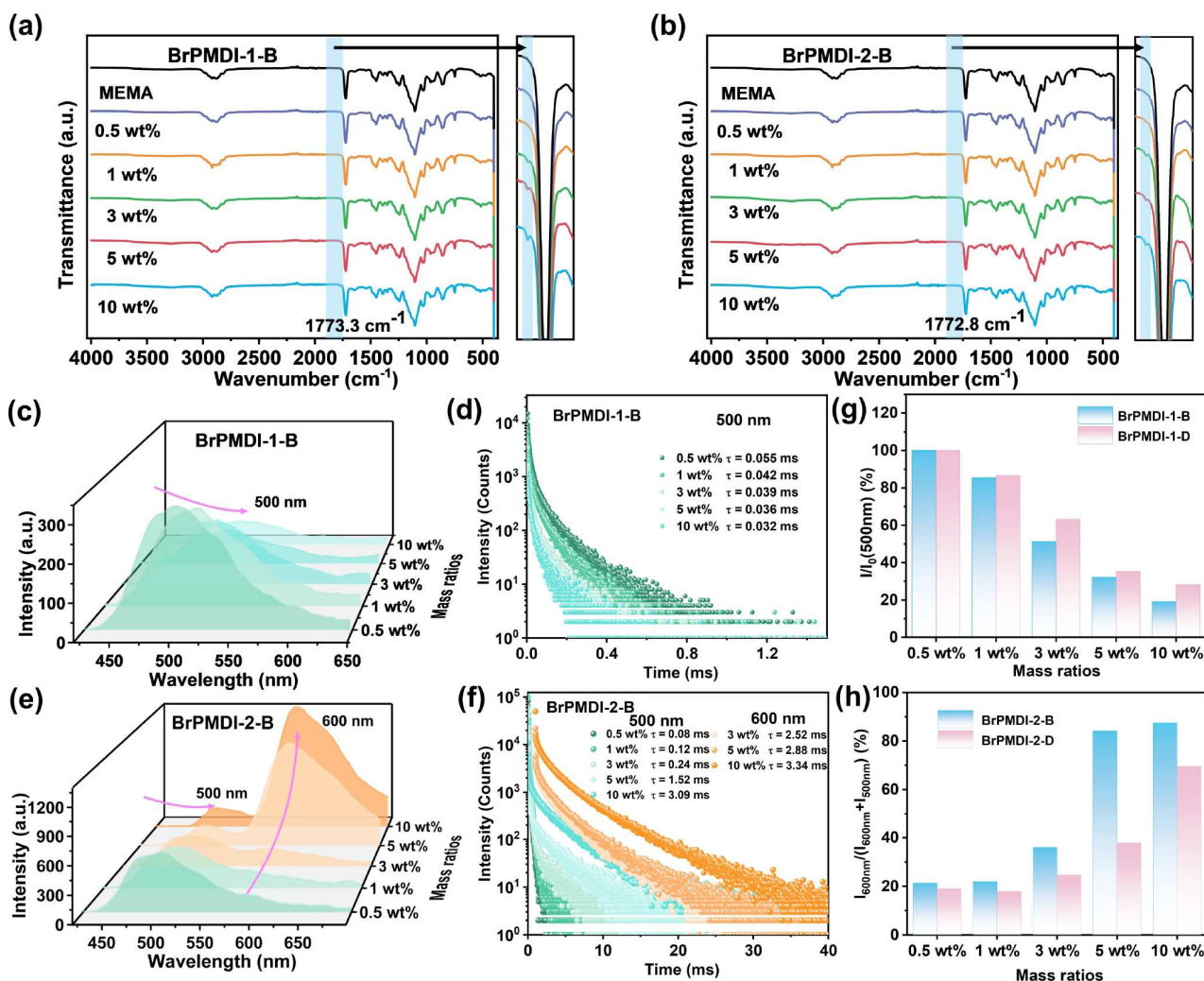


Figure 4 (Color online) (a, b) IR spectra; (c–f) delayed spectra (delay time: 0.05 ms) and time-resolved luminescence decay plots; (g) intensity ratios I/I_0 at 500 nm; (h) intensity ratios $I_{500\text{nm}}/I_{500\text{nm}} + I_{600\text{nm}}$ of elastomers containing various mass ratios.

aggregated emission peak was observed in elastomers. This arose from BrPMDI-1-D's high solubility, leading to over-aggregation and ineffective packing before reaching the concentration required for color change and resulting in reduced 500 nm intensity. For BrPMDI-2-B and BrPMDI-2-D, 600 nm intensity did not increase concomitantly with 500 nm attenuation, but a stage of exclusive 500 nm decrease was observed. Additionally, as shown in Figure 4g, h, BrPMDI-1-B exhibited a larger 500 nm intensity reduction ratio than BrPMDI-1-D. BrPMDI-2-B showed a higher 600/500 nm intensity ratio than BrPMDI-2-D. This demonstrated that the bridged structure promotes chromophore aggregation in elastomers. Additionally, steady-state and delayed emission spectra of the four chromophores in elastomers showed near-complete overlap (Figures S35–S38), confirming that copolymerization preserves their intrinsic pure phosphorescence.

3.4 Mechanoresponsive pure phosphorescence of bridged BrPMDI elastomers

To investigate the mechanoresponsive properties of the elastomers, the mechanical behavior of BrPMDI-1-B and BrPMDI-2-B elastomers with varying mass fractions was characterized. Stress-strain curves (Figure 5a, b) showed that increasing chromophore loading enhanced tensile strength at break but reduced elongation at break, attributed to chromophore-mediated crosslinking. For mechano-responsive tests, the minimum concentration was defined as that where BrPMDI-2-B exhibited a distinct luminescence color change. Based on maximum elongation at break, delayed emission spectra of 5 wt%-BrPMDI-1-B, 10 wt%-BrPMDI-1-B, 5 wt%-BrPMDI-2-B, and 10 wt%-BrPMDI-2-B were measured under different tensile strains (Figure 5c–f). At 400% tensile strain, 5 wt%-BrPMDI-1-B showed a 1.7-fold enhancement of 500 nm emission intensity, while 5 wt%-BrPMDI-2-B exhibited an increase in the 500/600 nm intensity ratio from 0.17 to 0.84. These results indicated that macroscopic tensile force is transmitted through polymer chains to aggregated chromophores, increasing interchromophore distance,

disrupting π - π stacking, and driving a gradual transition from aggregated to monomolecular states. For 10 wt% elastomers, reduced elongation at break (from higher chromophore loading) and enhanced aggregation hindered chromophore dispersion, leading to marginal changes in delayed emission spectra with stretching and loss of mechanoresponsive properties. Luminescence images under 365 nm irradiation, (Figure S39) and Commission Internationale de l'Éclairage (CIE) coordinates (Figure S40) corroborated these findings. Notably, 5 wt%-BrPMDI-2-B only shifted from orange to yellow luminescence, rather than the theoretical green emission of monomers. This was attributed to limited elongation at break, which prevented sufficient stretching to achieve a monomer-dominant state at maximum strain. Reducing chromophore loading improved elongation but precluded effective aggregation (below 5 wt%), failing to produce orange luminescence. Thus, an effective strategy is needed to enhance elastomer tensile strain without altering chromophore concentration or disrupting aggregation.

3.5 Optical and mechanoresponsive properties of supramolecular elastomers

Pr was synthesized, aiming to effectively increase the elongation at break of the elastomers by utilizing the pulley effect of cyclodextrins on the PEG chains. The influence of the Pr content on the tensile strain of the MEP elastomer matrix was investigated, and the effect of different mass fractions of Pr on the mechanical properties of the MEP elastomers was tested (Figure S41). The results showed that the addition of 0.5 wt% Pr exhibited the highest elongation at break enhancement rate (1.6-fold). Therefore, 0.5 wt% Pr was introduced into 5 wt%-BrPMDI-1-B and 5 wt%-BrPMDI-2-B to prepare the supramolecular elastomers 5 wt%-BrPMDI-1-B-0.5 wt%Pr and 5 wt%-BrPMDI-2-B-0.5 wt%Pr. As shown in the FT-IR spectra (Figure S42), a broad absorption peak appeared at 3313.2 cm^{-1} after the addition of Pr, originating from the stretching vibration of the hydroxyl groups on cyclodextrins, indicating that Pr was

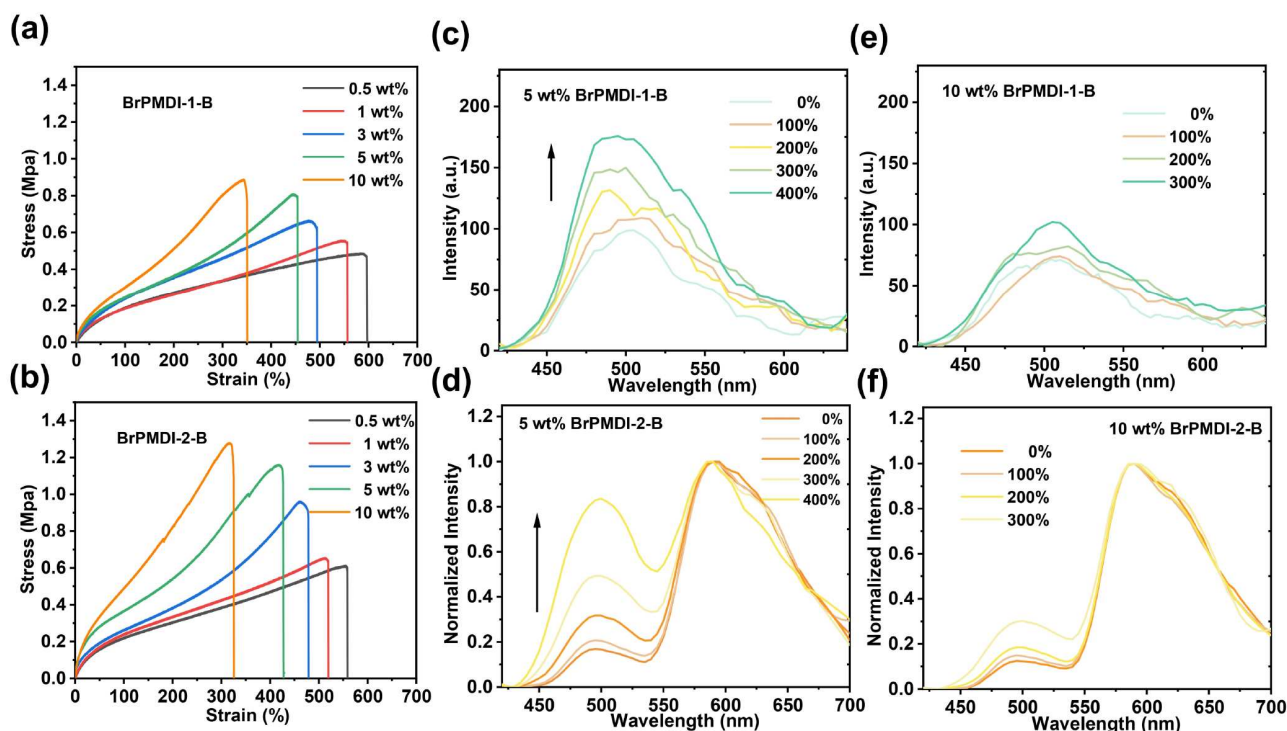


Figure 5 (Color online) Stress-strain curves of (a) BrPMDI-1-B, (b) BrPMDI-2-B elastomers containing various mass ratios; delayed spectra (delay time: 0.05 ms) of (c) 5 wt%-BrPMDI-1-B, (d) 5 wt%-BrPMDI-2-B, (e) 10 wt%-BrPMDI-1-B, (f) 10 wt%-BrPMDI-2-B under different strains during the tensile.

successfully introduced into the elastomers. The addition of Pr increased the maximum tensile strain of 5 wt% BrPMDI-1-B and 5 wt% BrPMDI-2-B by 1.2-fold (Figure 6a, b).

Meanwhile, both elastomers exhibited excellent fatigue resistance and maintained stable mechanical properties after multiple cycles of reciprocating stretching (Figure S43). In Figure 6c–f, the delayed emission spectra and lifetime decay profiles of the phosphorescent elastomers, both prior to and following the incorporation of Pr, maintained exceptional congruence. This observation underscores that the introduction of Pr exerted negligible influence on the aggregation behavior and photophysical properties of the chromophores within the elastomeric matrix, thereby safeguarding the superior mechanoresponsive capabilities of the phosphorescent elastomers. Subsequently, guided by the corresponding elongation at break values, the evolutions of the delayed emission spectra for the supramolecular elastomers (5 wt%-BrPMDI-1-B-0.5 wt%Pr and 5 wt%-BrPMDI-2-B-0.5 wt%Pr) under varying tensile strains were systematically interrogated (Figure 7a, b). It was revealed that the variation trend was analogous to that of the Pr-free counterparts, while the magnitude of the mechanoresponse was substantially amplified. For 5 wt%-BrPMDI-1-B-0.5 wt%Pr, the enhancement factor of the emission peak intensity at 500 nm escalated from the initial 1.7-fold to 2.2-fold. For 5 wt%-BrPMDI-2-B-0.5 wt%Pr, the ratio of the emission peak intensity at 500 nm to that at 600 nm under maximum stretching surged from the original 0.84 to 1.9. As illustrated in the luminescence micrographs of the two supramolecular elastomers irradiated with a 365 nm light source under different tensile strains (Figure 7c, d), at 500% tensile strain, 5 wt%-BrPMDI-1-B-0.5 wt%Pr exhibited a pronounced enhancement in luminescence intensity, whereas 5 wt%-BrPMDI-2-B-0.5 wt%Pr underwent a distinct visible transition from orange to green phosphorescence—an observation that aligns well with the CIE chromaticity diagram (Figure S44). Furthermore, the delayed emission spectra of the two supramolecular elastomers subjected to multiple cycles of reciprocating stretching (from 0% to 500%) (Figure 7e, f) demonstrated that they still maintain favorable

force-response performance after 5 repeated stretching cycles, showing remarkable mechanical reversibility and luminescence fatigue resistance of this system.

4 Conclusions

In summary, two types of stretchable pure phosphorescent materials featuring mechano-induced phosphorescence enhancement or mechanochromic phosphorescence were successfully fabricated through the radical copolymerization of bridged BrPMDI (BrPMDI-1-B or BrPMDI-2-B) with tailored ethylene glycol side chain lengths, MEP, and Pr. The two supramolecular elastomers achieved a stretch-induced 2.2-fold amplification of phosphorescence intensity and a distinct phosphorescence color transition from orange to green, respectively. In contrast to the elastomers without Pr, 5 wt%-BrPMDI-1-B with merely 1.7-fold phosphorescence enhancement and 5 wt%-BrPMDI-2-B exhibiting only a subtle transition from orange to yellow, the pivotal role of Pr in the stretchable mechanoresponsive system was underscored. This difference in force-induced response properties stems from the distinct phosphorescent properties of the aggregated states of force-sensitive phosphorescent molecules (BrPMDI-2-B exhibited orange phosphorescence in the solid state, compared to BrPMDI-1-B, which suffers from luminescence quenching). Raman spectroscopy and single-crystal X-ray diffraction corroborated that the side chain length modulates the photophysical properties of the aggregated state by altering the molecular packing motif and intermolecular distance. Concurrently, it was validated that the bridged architecture promotes the aggregation of chromophores within the elastomers and confers upon them intrinsic mechanoresponsive capacities. Benefiting from the augmented sliding mobility of cyclodextrin moieties within the Pr, the prominent pulley effect augmented the maximum tensile strain of the elastomers by 1.2-fold, which in turn potentiated the amplitude of the mechanoresponse. This study not only furnishes a novel and facile strategy for the molecular engineering of BrPMDI but also extends the realm

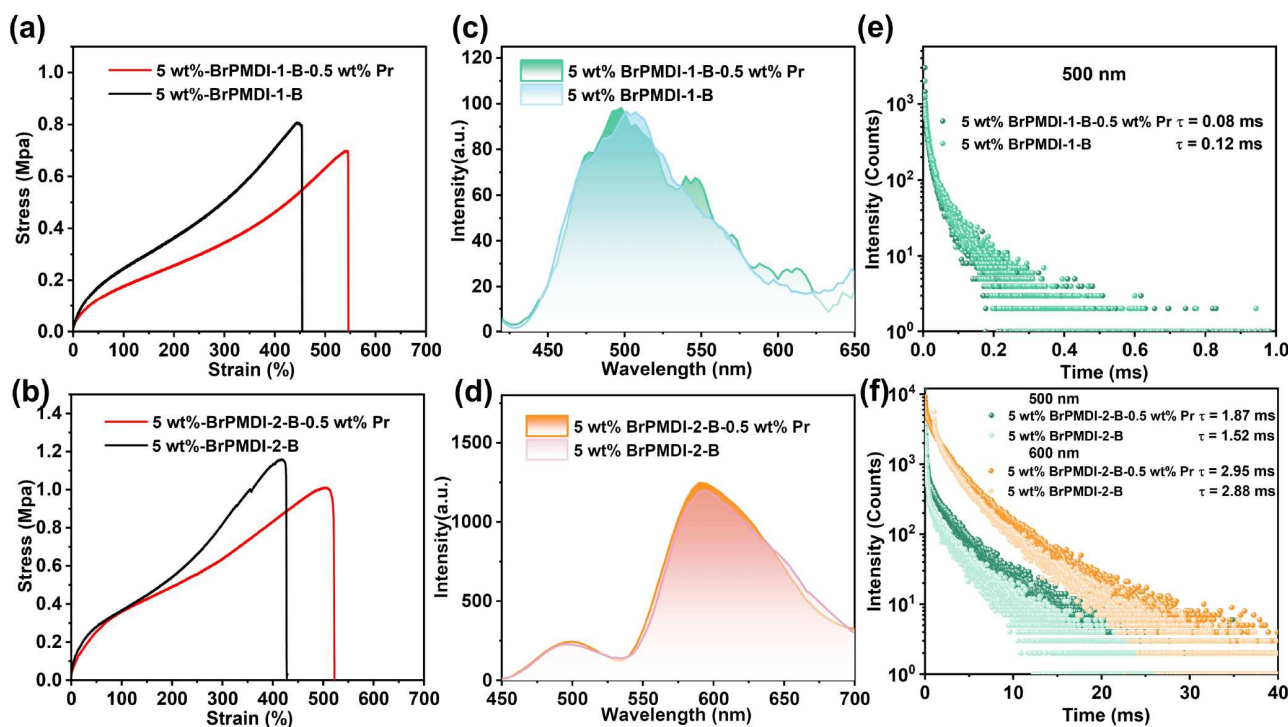


Figure 6 (Color online) (a, b) Stress-strain curves, (c, d) delayed spectra (delay time: 0.05 ms), and (e, f) time-resolved luminescence decay plots of phosphorescent elastomers with and without Pr for 5 wt%-BrPMDI-1-B and 5 wt%-BrPMDI-2-B.

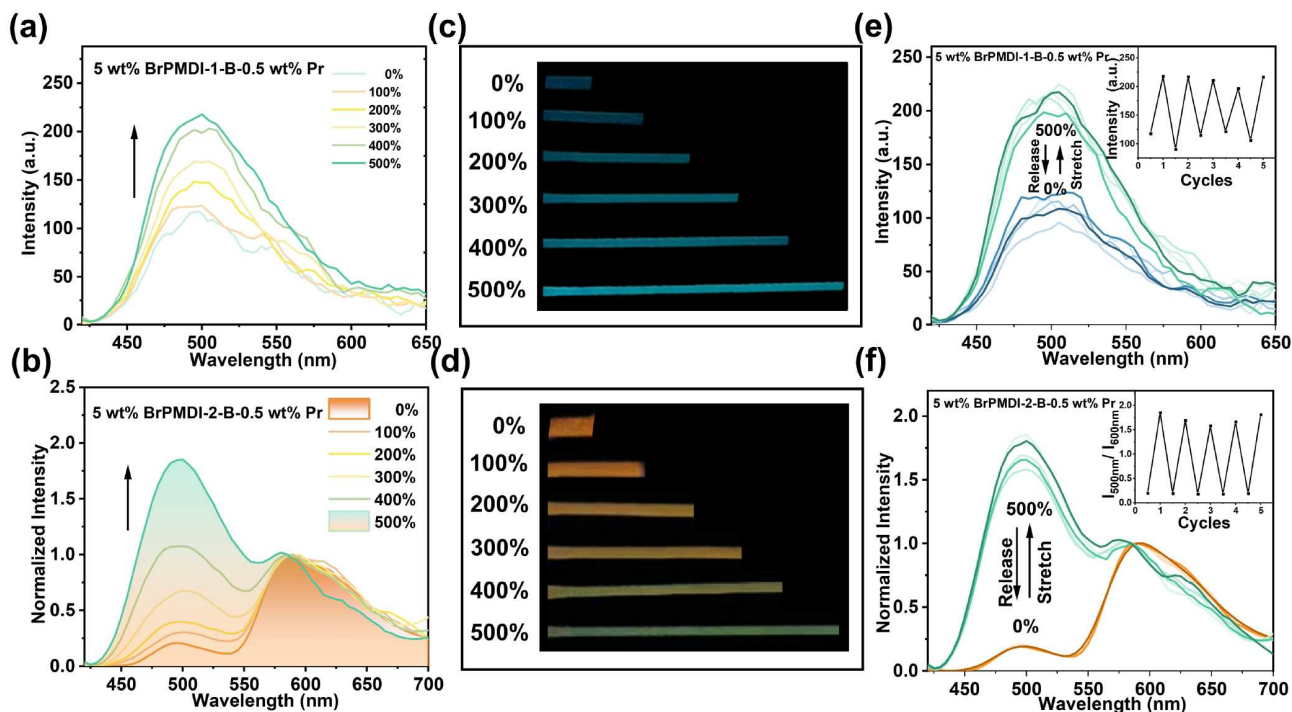


Figure 7 (Color online) Delayed spectra (delay time: 0.05 ms) of (a) 5 wt%-BrPMDI-1-B-0.5 wt%Pr, (b) 5 wt%-BrPMDI-2-B-0.5 wt%Pr; photographs of (c) 5 wt%-BrPMDI-1-B-0.5 wt%Pr, (d) 5 wt%-BrPMDI-2-B-0.5 wt%Pr with different strain under 365 nm UV light; delayed spectra (delay time: 0.05 ms) of multiple cycles of reciprocating stretching (from 0% to 500%) of (e) 5 wt%-BrPMDI-1-B-0.5 wt%Pr (inset: the intensity ratios I/I_0 at 500 nm on alternating 0% and 500% strain); (f) 5 wt%-BrPMDI-2-B-0.5 wt%Pr (inset: intensity ratios I_{500}/I_{600} on alternating 0% and 500% strain).

of stretchable mechanoresponsive materials to the domain of pure phosphorescence, thereby broadening the application landscape of mechanoresponsive materials to a considerable extent.

Conflict of interest

The authors declare no conflict of interest.

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Supporting information

The supporting information is available online at <http://chem.scichina.com> and <http://link.springer.com/journal/11426>. The supporting materials are published as submitted, without typesetting or editing. The responsibility for scientific accuracy and content remains entirely with the authors.

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